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Topics of the Month

British chemical industry—healthy and expanding

CHEMICAL manufacturers who attended the annual dinner of the Association of British Chemical Manufacturers last month heard a plea by Mr. W. J. Worboys, chairman of the Association and commercial director of I.C.I., for more exchange of experience between firms. He said he believed that this could be done without serious risk of the disclosure of confidential information and would benefit all. British manufacturers, he pointed out, cannot complain on the one hand if buyers of chemicals in overseas markets say they buy their chemicals from Germany because they know the German chemical industry is good and, on the other hand, refuse to publicise and demonstrate the excellence of their own products and the wide spread of their own chemical industry.

However, figures given by Mr. Worboys earlier in his speech show that the British industry is by no means unhealthy. Since 1948 it has expanded at about twice the rate of British manufacturing industry as a whole. During this period an additional £265 million has been invested in the chemical industry, this being approximately 12% of the total new investment in British manufacturing industry during this period. In 1953 the chemical industry exported 29% of its total production, and in the first seven months of 1954 chemical exports showed an increase of 16% over the corresponding period for 1953, as compared with an increase of 11% for manufacturing industry as a whole.

And, as an augury for the future, Mr. Worboys reminded his audience that the chemical industry's expenditure on research and development in 1952 was over £11 million, an increase of 32% on 1948. Further schemes to increase research and development work involve capital expenditure of more than £6 million and, when completed, will cause the annual expenditure by the chemical industry on research and development to rise to nearly £13 million, an increase of approximately 50% on expenditure in 1948.

In another speech, this time at the annual general meeting of the A.B.C.M., Mr. Worboys described the Association's efforts to stimulate productivity. Some useful work is being done by the six area committees which were set up to help firms to carry out the recommendations of the Heavy Chemicals Productivity Team which visited the United States, and also by the committee set up to stimulate, encourage and advise on the application of ' work study ' in the chemical industry.

Regrettably, Mr. Worboys was not able to make a statement regarding the recommendations in the Cremer Report on chemical engineering research; this is because the study of the problem that the A.B.C.M. have been making in co-operation with the British Chemical Plant Manufacturers' Association has proved exceptionally difficult. It would, perhaps, have proved relatively easy to recommend the

establishment of a research association of the normal D.S.I.R. pattern, but the investigators were not convinced that this was necessarily the right method for chemical engineering. Meanwhile, the subject is still being actively studied.

The German chemical industry grows again

BEFORE the war, Germany was the world's second biggest producer of chemicals, making 21.4% of the total. Today its share is only 5.2% compared with America's 42%, Russia's 11.1% and Britain's 7.9%. Thus, states Dr. W. A. Menne, President of the German Federation of the Chemical Industry, although the West German chemical industry's turnover is now D.M. 10.9 milliards, it has really 'scarcely recovered.' He quotes figures to show that Western Germany's return to the world market has by no means led to a sensational encroachment on other producers' spheres of interest and that no such encroachment is to be expected in the future. He points out that in 1938 the German share of the export markets was 24.4% and has now declined to about half of this, while the U.S. share has risen from 14.7% to 27.9% while Britain's share has insignificantly decreased from 15.6% to some 14%.

Turning to the future outlook for Western Germany's chemical industry, Dr. Menne refers to technical advances which have enabled a number of processes to be developed; for example, the pressure-refining of benzole, continuous coke vaporisation with oxygen to produce synthetic gas, the production of oxygen-thermic carbide (or the vortex motion calcinated method) for sulphurous ores. He also discusses the prospects for the foundation of a German petro-chemical industry, and points out that certain limits are imposed at the outset to development in this direction, since Germany will continue to be largely dependent on crude oil imports. For the time being it will be necessary to employ the available petro-chemical facilities for products such as plastics, synthetic fibres and solvents. However, at present the West German chemical industry is working to full capacity and its more immediate problem is to expand its production facilities, with the limited capital available, while at the same time devoting sufficient resources to research on new products and processes.

Dr. Menne's review of the German chemical industry appears as part of a *Financial Times* survey of German industries. This survey also includes an article on the German potash industry, which has undergone radical changes since potash was first mined in Germany. At the beginning of World War II Germany—originally the world's sole supplier—accounted for only about 67% of world production. About 20% (560,000 tons) were supplied by Alsace, about 10% by North America, and about 3%, or 84,000 tons, by Spain. After the war the picture changed once more. The United States has stepped up her production considerably. The splitting of Germany into two parts has meant that some 60% of the German production has come under the control of the Russians.

Since 1953 the Federal Republic and the Soviet Zone, each producing some 1,323,000 tons, have shared second place in world production next to North America. This has been done mainly by developing what were previously small mines in the area of Hanover, notwithstanding that in these mines the potash has to be raised from depths of between 1,500 and 2,700 ft., whereas, for example, the five American companies exploiting the Mexican deposits mine to a depth of 600 to 900 ft. only.

Titanium production by new process

AT a conference arranged by I.C.I. at one of their Birmingham factories, and attended by 50 of the senior technical staff of the British aircraft industry, it was confirmed that the process I.C.I. has developed for the production of titanium uses sodium in the reduction of titanium tetrachloride. Their main plant, which is in an advanced stage of construction and will be in operation within the next six months, will thus be the first large-scale production unit to operate a process other than that devised by Dr. Kroll, which came into prominence, particularly in the U.S.A., after the war.

The General Chemicals Division of I.C.I., who are responsible for making the raw titanium sponge, started research on this metal some years ago. It was eventually decided that reduction by sodium was the most promising process, especially for British conditions. Therefore, although I.C.I. erected and is working a titanium plant using the Kroll process (reduction of titanium tetrachloride by magnesium) in order to have some material available, most of its effort has been directed to the reduction of titanium tetrachloride by sodium.

This process in its essentials is not new. In fact, it was the method by which titanium was first isolated. It had been stated, however, that this reaction would be uncontrollable on a large scale, and the raw titanium sponge made by the sodium reduction of titanium tetrachloride would not have the necessary properties to allow good quality massive ingots to be made. I.C.I. research indicated that this was not necessarily true. A semi-technical plant was erected in 1953 and a larger pilot plant was erected and started operation early in 1954.

Such was the company's faith in the sodium process that it took the decision to build, and start design on, the large-scale plant late in 1953, well before the pilot plant started operation. This large-scale plant is being built in the North of England and, construction having gone according to schedule, it is expected that the first units will come into production in the first half of 1955 and that full production will be reached before August.

Concurrently with the General Chemical Division's work on producing raw titanium, extensive research and development work has been done by I.C.I.'s Metal Division on processes for melting and fabricating titanium and its alloys. A pilot plant at one of their Birmingham factories has been melting raw titanium sponge for some time past at the rate of 150 tons p.a. and converting it into ingot, forging stock, and various fabricated products for the aircraft industry. A major plant for melting and preliminary fabrication of titanium and its alloys will be completed early next year.

The capacity of the full-scale plants which are being erected is 1,500 tons p.a. Though this capacity may seem small compared with the announced intentions of the American makers, it is not much less than the amount of titanium sponge actually produced in America in 1953 (2,250 short tons), and the plants have been designed so that they can be expanded when required.

Chemical, chemical engineering and plastics exhibition in Paris

IN our December issue we shall be publishing an illustrated review of the exhibits to be seen at the 3rd Salon de la Chimie et des Matières Plastiques, which is being held in Paris from December 3 to 12. The scope of this exhibition has been considerably increased and this year there will

Comical Engineering Terms

"ALCOHOL RECOVERY"

be four main sections: Chemical Engineering, Chemical Products, Laboratory Equipment, and Plastics Industry. Exhibits will include chemical plant, chemicals, constructional materials, processes and instrumentation, and in the Plastics section, raw materials, machinery and manufactured articles. We hope, with the co-operation of exhibitors, to make our survey of these exhibits as comprehensive as possible, and there should be many new developments to interest our readers.

American coal research

DWINDLING supplies of coal, the advent of atomic energy, the rapid growth of petro-chemical industry, new carbonising techniques, natural gas developments; these and other factors tend to obscure the future of coal as a fuel and as a raw material for processing, so that today there are many conflicting opinions as to the role it will play 20, 50 or even 100 years hence.

To keep a balanced view of this complicated question, we can only be guided by current trends in industrial activity and in research. Thus it is interesting to learn that the Koppers Co. in America is directing its research increasingly to chemical applications of coal, since the management is uncertain, in the long term, of the future of coke ovens. In particular, work is in progress on the partial hydrogenation of coal for the production of chemicals rather than fuels, on the lines established by the Carbide and Carbon Chemicals Corporation and described in CHEMICAL & PROCESS ENGINEERING, 1953, **34** (6), 165-168. Research into complete gasification, particularly by the fluidisation of coal in steam and oxygen, is also being carried out.

By contrast, at another American research centre—the Illinois State Geological Survey at Urbana—one of the chief interests of the Chemistry Department lies in blending coal for coke making; in this connection a very neat 17-in. moving-wall coke oven has been installed for the detection of dangerous coals and assessing blends. Both these establishments were visited towards the end of 1953 by Dr. I. G. C. Dryden, of the British Coal Utilisation Research Association, who spent some five weeks in the U.S.A. In a recent issue of the Association's *Quarterly Gazette*, Dr. Dryden's experiences are briefly recorded and a fair picture of trends in American coal research is given. Among laboratories he visited were those at the Bruceton station of the Bureau of Mines, which house large-scale experimental plant for coal hydrogenation and the Fischer-Tropsch hydrocarbon synthesis. The station is recognised as one of the best equipped of its kind in the world.

Another research station visited was a small one maintained by the Bureau of Mines at Denver, Colorado, where pilot-plant research is carried out by a staff of about 20; only a few projects are undertaken at a given time, and these all have definite technological objectives. One of the most important is the fluidised carbonisation of lignite prior to its combustion as a pulverised fuel; this research is conducted under a co-operative agreement with the Texas Power and Light Co., and is thus partly supported by private industry as well as by the Bureau of Mines. Very high yields of tar—12 to 14% by weight of the dry ash-free lignite—are obtained by carbonising the lignite to 900°F. in 10 to 15 min.

At the laboratories of Pittsburgh Consolidated Coal Co. work on coal pyrolysis is in progress, as well as on the development of tests for predicting tar yields in fluidised beds and for determining the plasticity of heated coals; there are also pilot plants for the hydrogenation of tar and fluidised carbonisation. The Disco plant for low-temperature carbonisation belongs to this company; circumstances making this plant economic are peculiar to the locality and there seems little prospect of its extension elsewhere.

What is industrial engineering?

THE last of the Reports of the series of 67 British Productivity Teams that have visited America has now been published.* The Industrial Engineering Team, which compiled it, was given the most comprehensive brief of all: to investigate the dynamic and aggressive American approach to improvement of methods of manufacture and to continuous reduction of operating costs. The report is correspondingly all-embracing and discusses such subjects as financial budgeting and control, design, planning and control of production, utilisation of research, plant layout, materials handling, time study, motion study, tool design, incentives, job evaluation and industrial relations; it also considers appropriate training for the industrial engineer.

The term 'industrial engineering,' although quite well known in the U.S., has only recently been in common use in Britain. Like most of the terms used in the field it applies to, it awaits definition. For the purpose of its report, the team defines the function as follows:

Industrial engineering is the application of basic scientific and engineering knowledge and certain analytical techniques to the methods of operating an organisation in industry and other appropriate fields. It requires investigation into the economic advantages of one policy as against another and implementation of the most suitable procedure for raising productive efficiency. To achieve this end, performance and costs need to be checked constantly.

Industrial engineering, then, is a staff activity of manage-

*'Productivity Report on Industrial Engineering,' British Productivity Council, 5s. net.

ment and is concerned with decisions on what to produce, the design of the product, standardisation, the influence of cost analysis on methods of production, the investigation of these methods, planning and control of production, calculation of what is the most efficient machinery for the job, layout of plant and handling of materials, design of tools, maintenance of plant, time study, motion study, incentives, method of calculation of wages, suggestion schemes and industrial relations. It requires consideration to be given to the communication of new ideas among senior and junior managers, among supervisors and to and from workers on the shop floor. It also requires careful regard to the well-being, attitudes and opinions of workers.

In the United States, industrial engineering as defined above is carried out by various types of 'hyphenated engineers' as they have been so aptly described: management-engineer, methods-engineer, process-engineer or production-engineer. But there is no common meaning for any one of these terms. The function is often carried out by men with another title—by a controller, by an executive vice-president, by a plant manager or his assistant.

Scale models help chemical plant designers

THE news that in America an oil company is providing schools with model kits from which pupils can make up a miniature oil refinery, thus learning the significance of the different units and how they work together to produce the various refinery products, makes us look back wistfully at our own schooldays. We ourselves were never given anything more exciting than modelling clay, which did not lend itself, unhappily, as a material of construction for cat crackers or fractionating columns.

However, it seems that Americans are using models of one sort or another for purposes other than initiating the young into the wonders of oil refining. In particular, they are being used to assist in the design and construction of chemical plants. For, caught between the demand for lowering engineering costs, good design for new complex plants, and a shortage of competent engineering talent, today's designer of chemical plants is turning to the use of the scale model as a three-dimensional planning tool.

Ten years ago chemical plant models were expensive toys. They were built from final drawings and attempted to duplicate every complex detail. The cost was high, the timing late. But recently techniques for building models have been standardised and the showing of unnecessary details has been dropped. Describing the construction and use of these models, a recent article in *Chemical Engineering* draws a distinction between the 'training model,' which, built after design, is useful for detecting interferences and for training operating labour, and the engineering model, which is built ahead of, or parallel with, the blueprints. The engineering model costs about twice as much as the training model, because it is being continually revised; however, it not only serves to detect errors at an early stage, but tends to prevent errors creeping into the design in the first place.

A preliminary engineering model, built from flowsheet data, is followed by a basic model, without waiting for steel and equipment details. Design is flexible at this stage and equipment on the model is movable. The third phase involves piping design. All important pipes (depicted with thin brass wires), valves and instruments are shown. Piping drawings are not made until after the model has been reviewed and corrected by operating, maintenance and construction personnel. In the fourth phase, which is optional, the model maker strips out the fine wires and, working from the drawings, installs 'piping' to scale. This serves as a check on the drawings.

The model is sent to the construction crew and is used by subcontractors in preparing their bids and by construction people in planning building schedules. Finally, the model can be used to help in the training of operating, maintenance and supervisory personnel. It is estimated that the cost of the engineering model is small compared with the savings in piping drawings, pipe and fittings, and subcontracting work.

Britain to make new type of cationic and nonionic chemicals

A PLANT which is being built at Littleborough, near Rochdale, Lancashire, will carry a step further the introduction into this country of a new line of chemicals pioneered and developed by Armour & Co. in Chicago. It began here when Hess Products Ltd. started to produce the *Distec* range of fractionated fatty acids a few years ago. The new venture is a logical development of this.

Nearly all the new plant will be of British manufacture and will be erected and operated by British labour. It will be adjacent to the existing Hess Products' fractional distillation unit, which has been producing *Distec* fatty acids since 1950. Fatty acids will be pumped across to the catalyst plant now under construction, where they will be contacted with ammonia. Operation will be continuous and completely automatic, yielding high-grade aliphatic nitriles. As the nitrile issues from the plant in continuous flow it is passed to the hydrogenation section, where it is turned into the primary amine. Depending on the type of fatty acid used, a whole range of these amines can be produced.

By a modification of this process, other amines can be produced, including the diamines of the general formula $RNH\ C_3H_8\ NH_2$. By reacting any of the amines with an organic acid, water-soluble salts are obtained which have high surface activity. Amongst the new chemicals will therefore be the *Armacs* and *Duomacs*, which are the water-soluble salts of the amines and diamines respectively. The amines are also used as starting materials for a wide variety of organic chemicals. They are, for instance, quaternarised to give a range of bactericidal and substantive products. They are the basis for certain ampholytic chemicals, alkyl morpholines and many others, including ethylene oxide compounds.

When completed the total plant will have cost in the neighbourhood of £250,000. This expense will be met from the companies' own resources and no fresh capital will be needed. Hess Products have recently signed a contract with royalty payments to the American company, for which they have obtained the exclusive manufacturing rights and know-how for the new process and also any new developments in this field which might be discovered through the Armour research organisation. Sales and distribution of the new chemicals, both for the home market and for export, are exclusively in the hands of Armour & Co. Ltd., Chemical Division, London.

Hess Products Ltd. are associated with the old-established Leeds firm of Adolph Hess & Brother Ltd., which has been in the distillation business for 80 years. Also connected with Hess Products Ltd. is the well-known Leeds chemical firm of Brotherton & Co. Ltd., who have a considerable interest in this development. This extension of a new industry in East Lancashire continues the recent national policy of getting a wider diversity of interests in industrial areas.

AUTOMATIC CONTROL OF DISTILLATION PROCESSES

I. General Considerations

By A. Pollard, B.Sc., A.R.I.C., A.M.I.Chem.E.

Much of the remarkable development in the techniques of industrial distillation during the past 15 to 20 years can be attributed to the application of automatic control to these processes. In this article, the first of two on the automatic control of distillation plants, the basic general problems of plant control are discussed with reference to the continuous fractionating column. The effects of changes in feed conditions and the influence of the column design on the methods of control are considered, and typical control systems for different cases are outlined.

THE past two decades have witnessed remarkable changes in the practical aspect of the distillation process, mainly as a result of the developments in the petroleum refining and petro-chemical industries. The development of the alkylation process for high-octane fuels, followed in rapid succession by such other processes as catalytic cracking, hydroforming, synthetic rubber and the production of a wide range of other chemicals from petroleum residues, has placed greater emphasis on the precise fractionation of feed stock and products. In addition to a progressive increase in column size, both in the number of plates and in diameter and capacity, there have also been decided changes in the manner of utilisation.

One of the contributing factors in the successful development of these complex processes has been the use of automatic control of the distillation process. It can be safely said that in many cases these processes could not be operated successfully without the use of automatic control. But even in the simplest distillation plant the use of automatic control, *when properly applied*, cannot be other than beneficial.

Any continuous fractionating column when operating smoothly is in a state of continuous dynamic equilibrium. The feed enters and the products are withdrawn at constant rates; the supply of heat to the reboiler and of cooling medium to the condenser are constant. The composition of the material in the system also remains constant and, correspondingly, the pressure and temperature at any point are constant. Once attained, this state of equilibrium must be maintained and adjustments must be made slowly and carefully and only when necessary. Automatic control can assist considerably in the maintenance of the best possible equilibrium conditions, despite the unavoidable disturbances which arise from outside the actual distillation plant, and with which even the best still operator has difficulty in coping. Changes in the quantity and quality of the feed, for example, require the adjustment of several other variables and the imposition of an entirely new equilibrium in the plant.

The distillation process, however, can present a rather discouraging picture to the engineer or designer faced with the problem of instrumenting such a process for automatic control. This is due mainly to the number of process variables involved —even in the case of a simple fractionating column—and their interdependence upon each other, and the number of different ways in which control of a particular variable can be accomplished. The method of application has been only too frequently that of trial and error or the duplication of a similar system. But the trial and error method can be expensive, and duplication of an existing application, which in itself may be quite efficient, by no means guarantees equally efficient results. In the present article the general problem of distillation plant control will be discussed and a logical approach to the automatic control presented so that the instrumentation can be wisely and economically planned.

Basic problem

The basic general problem of control is the same irrespective of the application or whether the control is to be manual or automatic. To enable a plant to give its designed performance, certain conditions specified in the design must be fulfilled and maintained by the plant operators or by the automatic control system. In general the requirements will be that in certain parts of the plant the principal process variables, *e.g.* temperature, pressure, rates of flow, liquid levels, etc., should be kept constant or within specified limits, or alternatively may be required to be varied with the state of the feed supplied to the plant. A distinction can be drawn between a constant feed, which is invariant in quantity and composition, and a variable feed, which can vary in both quantity and composition.

Each of the principal process variables —rate of flow, pressure, temperature and liquid level—can be controlled directly or indirectly by regulating the flow of material or energy into or out of the plant. The regulating unit is most frequently some type of valve, but can also include the speed regulation of a prime mover operating a pump or compressor.

To enable the design conditions of the plant to be maintained, the control system will thus require a certain number of valves, each of which will regulate an actual process stream in the plant. There may be fewer control valves than process streams, *i.e.* certain streams may be 'free' or uncontrolled, but logically there cannot be more.

It follows that, if more design conditions have been specified than there are controllable streams in the plant, then either the design conditions are not independent or the plant is uncontrollable.

In the majority of cases all streams that form an integral part of the flow sheet of the process will require to be regulated, and the number of design conditions should then be the same as the number of streams. The initial problem of plant control will then be the identification of the controllable streams and the design conditions, and the correlation between the two. The latter problem, the choice of which design condition to regulate a particular process stream, is often difficult in practice, and particularly so in distillation control, since the process variables are not generally independent of each other. The various reactions following a change in the flow of a regulated stream may have several effects, and it is not always possible to distinguish immediately the primary reaction from a secondary or even tertiary effect.

Control of three variables

This last point can be well illustrated by the example cited by M. Ruheman (*Trans. Inst. Chem. E.*, 1952, **30**, 125) of the partial evaporator, a plant unit with three controllable streams, which can be regarded as a primitive type of distillation apparatus without fractionation. Briefly the purpose of the unit is to separate a stream of liquid

mixture into gaseous and liquid components by partially evaporating the more volatile constituents by a suitable heating system. Three design conditions can be specified, the temperature and pressure, which are determined by the vapour-liquid equilibrium of the mixture and the particular separation desired, and the liquid level in the vessel which must be maintained within limits sufficient to cover the heating element and provide an adequate disengaging space above the liquid. There are also three controllable streams in addition to the liquid feed—the gaseous and liquid products, and the flow of heating medium. The feed must be regarded as 'uncontrollable' in the sense that it is not regulated by the design conditions within the evaporator; the supply may be controlled externally, at a constant or variable rate, or it may be 'free,' *e.g.* the product of a preceding stage in the process.

To maintain the three design conditions it should be adequate to control by valves regulating the flow of the three process streams. In view of the nature of the design conditions, it is reasonable to assume that, for automatic control, three instruments will be required and each will operate the control valve in one particular stream. The instruments will be logically a temperature controller (*TC*), a pressure controller (*PC*) and a liquid level controller (*LLC*). The control valves will be the vapour outlet valve (*VV*), the liquid outlet valve (*LV*) and the heat supply valve (*HSV*), as illustrated in Fig. 1.

However, there are six possible combinations between three controllers and three valves, *viz.*

(1)	(2)	(3)
PC—VV	*PC—VV*	*PC—LV*
TC—HSV	*TC—LV*	*TC—HSV*
LLC—LV	*LLC—HSV*	*LLC—VV*
(4)	(5)	(6)
PC—LV	*PC—HSV*	*PC—HSV*
TC—VV	*TC—VV*	*TC—LV*
LLC—HSV	*LLC—LV*	*LLC—VV*

In this relatively simple example, the first arrangement is the obvious logical choice, the reason being that in this case each control reaction will immediately counteract the impulse or deviation initiating the reaction. It is important then to distinguish between the primary and secondary effects of the controller reaction, particularly in more complex cases. Taking, for example, the vapour outlet valve (*VV*), the primary effect of opening this valve is to lower the pressure in the vessel by allowing more vapour to be withdrawn. It is then logical to use this valve as a pressure control. But there are also two secondary effects due to the fall in pressure; the increased evaporation due to the production of more vapour will cause a fall in the temperature and a reduction in the liquid level. The valve could then be used, theoretically at least, for the control of either the temperature or the level. It can

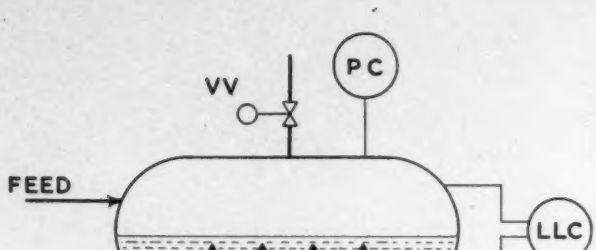


Fig. 1. Control of the partial evaporator.

in fact be demonstrated that all the above arrangements of controllers and valves will 'work,' but with varying degrees of efficiency.

To judge a particular arrangement it is useful to consider the plant running under the design conditions, and to assume a disturbance, *i.e.* a deviation from any one of the desired conditions, to occur. The disturbance provides the impulse to the control system, which will seek to oppose the deviation of the particular variable considered. So long as this ultimately occurs, the control system will work, but the response can only be considered efficient if

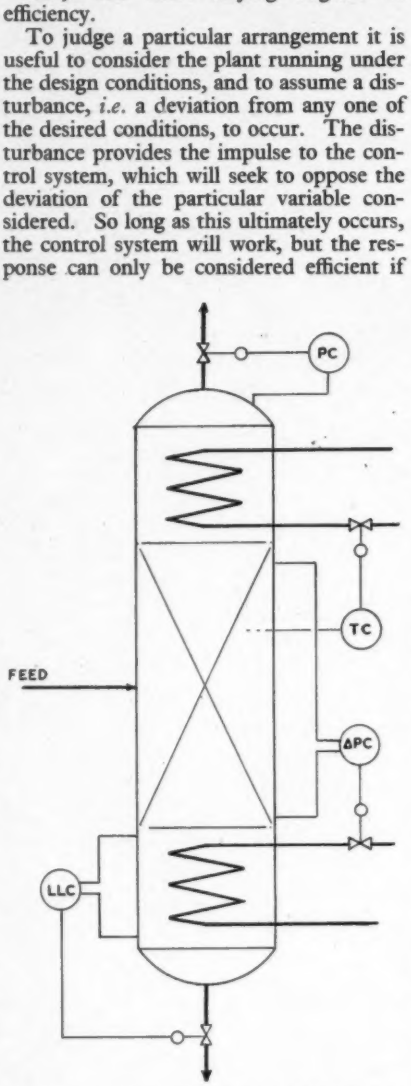


Fig. 2. Continuous column with integral condenser and reboiler.

the control reaction opposes the deviation directly and not at second- or third-hand.

Considering the last arrangement above, which represents one of the two worst possible cases, an increase in pressure would close the heat supply valve and produce initially a decrease in temperature. In the absence of the other controls this would reduce the rate of evaporation and ultimately lead to a fall in pressure. However, the temperature controller would react to the fall in temperature by opening the liquid outflow valve and so reduce the level, and the level controller would respond by opening the vapour outflow valve. The pressure is thus reduced by operation of the proper valve, but only by a third-hand reaction. It can be similarly demonstrated that a change in temperature or level has similar results, and that the fourth arrangement above also gives only third-hand reactions. Of the remaining arrangements, only the first gives an immediate counter-action to a change in any one of the variables, the other three requiring secondary reactions in the case of two variables.

It is also of interest, in view of the importance in the application to the fractionating column to consider the effect of changes in the quantity and composition of the feed in this simple case. This will also show whether the specified design conditions are in fact adequate to yield the required products under the new conditions.

It will readily be seen that in the present case the design conditions are independent of feed variations. If the rate of flow is increased without changing the composition, then new equilibrium positions of the control valves will be established to maintain the original conditions of level, pressure and temperature. The level will initially rise, the temperature will fall due to the increased throughput and the pressure will rise due to the increased rate of evaporation. Thus all three valves will open simultaneously. If the rate of feed flow remains constant but the composition changes, say by an increase in the more volatile constituents, then again new

equilibrium positions of the valves will be found which will yield more gaseous and less liquid products with the original design conditions.

Process variables in the fractionating column

In a complete fractionating column, four external process streams can immediately be distinguished apart from the feed—the overhead and bottoms product, and the streams carrying heating and cooling media to the reboiler and condenser respectively. There are also the two important internal streams, the vapour flow up the column and the liquid flow down the column, both of which are virtually independent of the feed and product flow rates and are governed by the reflux ratio. With a high reflux ratio the internal streams may be very much greater than the flow of products. The internal and external streams, however, are clearly not independent of each other, since the heat supply to the reboiler effectively determines the vapour flow up the column. The liquid flow down the column is determined by the flow of cooling medium to the condenser in the case of a column with an integral reflux condenser, or an external condenser producing only reflux. In the latter case, and also in the case of total condensation of the overheads, the reflux return can be regulated directly and becomes effectively a fifth external stream.

The number of design conditions, and hence the number of controllers required, will depend to some extent on the arrangement of the various parts of the column, as briefly mentioned above. To preserve the heat and material balance, at least two conditions are essential:

(*a*) The pressure at some point must be held constant; and

(*b*) The liquid level in the reboiler must be maintained within certain limits.

These conditions apply in all cases, but further additional conditions may be necessary to preserve the heat and material balances. If, for example, the column is fitted with vapour-liquid separators (*e.g.* a 'reflux drum' after an external condenser), or if the condenser is cooled by the evaporation of a refrigerant (a 'two-phase' condenser), then additional liquid levels must be maintained as design conditions.

These conditions for heat and materials balances are clearly necessary, but in themselves are not sufficient to ensure that the column will yield products of the specified compositions. Two further conditions have to be imposed in all cases:

(*a*) The reflux ratio must be kept constant or at least above a minimum value *or* it must be adjusted (depending upon feed conditions) to give an overhead product of the required composition; and

(*b*) The boil-up rate must be adjusted in conjunction with the reflux so that the overhead and bottoms products are separated in the same relative proportions that the two products are introduced in the feed.

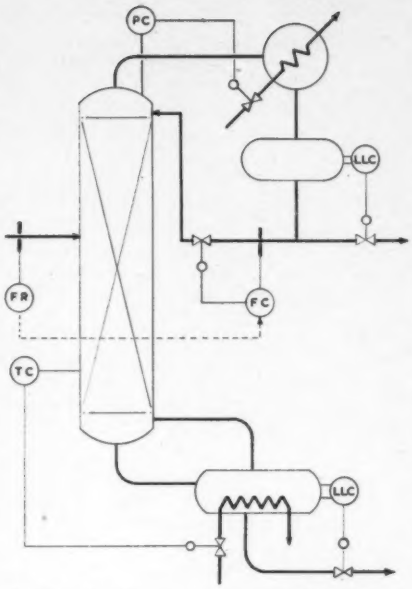


Fig. 3. Continuous column with external condenser and reflux drum.

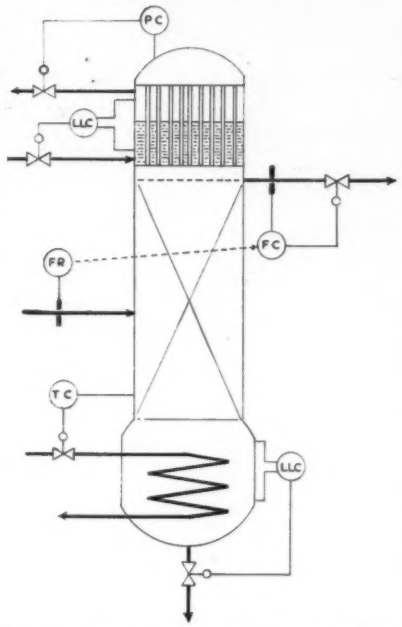


Fig. 4. Continuous column with integral two-phase condenser.

Neither of these last two conditions can be expressed directly as variables which can be controlled by the regulation of process streams, but it is not difficult to find a number of controllable variables, two of which can be used to maintain these conditions.

The simplest example for illustration of these points is presented by a fractionating column with integral single-phase condenser, as shown in Fig. 2. Here there are four controllable streams and four design conditions. Four controllers should then be adequate. If the first two conditions, the pressure and reboiler level, are maintained by regulation of the product streams, then the second two conditions, the reflux and the boil-up, must be maintained by regulation of the remaining external streams, the condenser cooling and the reboiler heat supply. Two variables must be selected which effectively are determined by the reflux ratio and the boil-up; logically these will be the temperature at a point in the column and the differential pressure across the column. It is not, of course, possible in this case to regulate either of the internal streams directly without materially altering the design of the apparatus.

It should perhaps also be noted in passing that, if condensing steam is used as the heating medium, then a further controller is theoretically required to 'maintain' the steam condensate level. This is the duty of the steam trap, which is not usually regarded as a process controller, but the case is exactly analogous to two-phase cooling of the condenser, as in Fig. 4.

The use of an external condenser with reflux drum requires a further controller on the level in the reflux drum. The reflux return and the product take-off streams are now both external to the column and can be regulated directly, as is illustrated in Fig. 3, in which the reflux flow is measured and controlled at a value reset by the flow of feed. Control of the boil-up is based on a plate temperature below the feed plate. In Fig. 4 a two-phase integral condenser is used, the supply of refrigerant is regulated by the liquid level and the vapour outflow by the column pressure. A liquid product is taken off the top plate at a controlled flow, reset by the feed flow.

Effect of variations in feed conditions

In the preceding discussion it has been tacitly assumed that the feed to the column is constant in quantity and composition. However, in the case of a fractionating column—and also the stripping and rectifying sections if used separately—the design conditions are not independent of variations in the feed supply. This is due to the existence of pressure and temperature gradients throughout the column, which are determined by the particular equilibrium conditions existing at the time. Since it is possible to control the pressure or temperature at one point only, the values of the pressure or temperature at other points are then dependent variables and as such are uncontrollable.

The pressure gradient in the column is due to the static head of the liquid on the plates and the resistance to the flow of vapour up the column and, whilst the former is effectively constant, the latter varies with the vapour velocity. At a constant re-boil ratio the pressure dis-

tribution will thus vary with the load on the column, *i.e.* with the rate of feed supply.

It will then be apparent that if the feed rate is increased—without at the moment considering the effects of any change in composition—the pressure differential over the column will be increased. The pressure, however, can only be controlled at the one point, which is generally at the top of the column or at the condenser for reasons of convenience, so that an increase in pressure will be evidenced at all points below. This will in turn lead to an increase in the boiling points of the liquid on the plates below.

It is, of course, usual in practice to take the temperature at an intermediate point in the column as an indication of the composition of one or other of the products for control purposes in both manual and automatic systems. Thus the result of the increased feed rate will be to increase the temperature but without any change in composition of the liquid on the plate. The temperature controller will react logically to restore the original temperature, but can only do so by changing the composition of the liquid on the plate and thus also changing the composition of the products. Fortunately in practice the change in pressure is often not sufficiently large to cause a significant change in the product composition, but the effect can be minimised by providing a means of pressure compensation in the temperature measuring device which effectively resets the control point of the temperature controller as the pressure varies. Alternatively the pressure and temperature can both be controlled at the same plate, although this is not always practical.

The temperature gradient in the column is due to the differences in the boiling points of the liquids on the successive plates and is determined, for a given pressure gradient, by the composition gradient between the two products. Changes in the composition of the feed require the establishment of an entirely new equilibrium, with new composition gradients above and below the feed plate. To maintain the desired separation it is necessary to adjust the reflux ratio, but whether the desired separation can still be accomplished is essentially now a question of column design, in particular whether a sufficient number of plates now exist above or below the feed.

It is conventional design practice to provide a number of 'spare' plates at each end of the column which under the normal feed conditions play no part in fractionation but simply carry liquid of the composition of the overhead or bottom product. As the feed composition varies, these additional plates effect further fractionation at one end or other of the column, but ultimately a feed composition will be found at which there are insufficient plates either above or below the feed plate to effect the desired separation. This point represents the limit of the column design, and must also be the limit of the control system, since the automatic controls cannot be expected to correct for faulty design.

It is extremely doubtful if any successful control system can be devised which can counteract the overloading of the column in this way by excessive variations in the feed composition. It is necessary to ensure that the column design is adequate to give the required separation under the worst possible feed conditions; the control system can then be designed to take care of variations in the feed.

Assuming that the column design is adequate to yield the desired separation, then the situation is very similar to the previous case, in that the temperature distribution will again be altered, but now due to the changes in the liquid composition on the plates. Temperature control from an intermediate plate will then again call for a resetting of the temperature control point. There are several ways in which this can be done, both manually and automatically, but all are complicated. Whether this refinement is necessary will depend upon the accuracy of separation desired and the amount of variation in the feed composition.

Control by material balance

It will be appreciated from the preceding discussion that the problem of fractionating column control is much simplified if the feed is invariant in quantity and composition. For economic reasons alone it should always be the aim to operate a column at a constant optimum loading which is usually just below the maximum at which the column floods. Whilst this can frequently be ensured, the control of composition is rather more difficult unless the feed is drawn directly from stock.

Under conditions of constant feed, however, it should not be necessary to base the control on the product compositions, since if the design conditions are fulfilled the plant should yield products of the desired composition. Control can then be based on a strict material balance. The amount of overhead product is a fixed proportion of the feed; hence, if the latter is constant, a flow controller can be used to remove the required proportion of product, the remaining overhead condensate being returned to the column as reflux. The material balance is preserved by a simple level control at the base.

With a constant feed it should also be possible in theory to control also the heat input and cooling supply by flow controls. Due to the possibility of erratic boiling and the fouling of the heat exchange surfaces it is preferable to utilise column variables which are directly influenced by these two streams. The heat input is then frequently controlled by the pressure differential across the column and the condenser cooling by the column top pressure or the level in the reflux drum.

If the feed is variable in quantity only it is still possible to use material balance as a basis of control by varying the take-off of overhead product in strict proportion to the flow of feed by the use of a cascade or ratio flow control. In this event it would also be preferable for economy to control the heat input also on a rate of flow basis, reset by the feed flow control.

Control by temperature

When the feed varies in composition the relative proportions of overhead and bottom products in the feed will change, and control on a material balance, withdrawing a fixed proportion of feed as overheads, will not then yield products of the required composition. Control must then be based on the measurement of the composition of one of the products. Due to the new equilibrium conditions which must now be set up within the column and to limitations of the column design, the composition of the second product cannot usually be maintained exactly, but some variation must be permitted as the feed composition varies.

In all but a relatively few cases the measurement of the product composition cannot be preformed directly, and generally the temperature at some point in the column is taken as indicative of composition. This method must, of course, be indirect, and the temperature-composition relationship is subject to pressure variations. It would be preferable to use a direct measure of composition by some method of physical analysis, but unfortunately such methods are not at the moment of sufficiently wide application in the field, particularly with multi-component mixtures.

With temperature control, the method is one of regulating either the reflux return or the heat supply to the reboiler to give the specified overhead or bottom product respectively. In the first case the point of temperature control will be above the feed plate and the heat input will be maintained constant or regulated by the flow of feed. In the alternative case, the temperature will be controlled at a point below the feed plate and the reflux return held constant or regulated by the flow of feed, at a value consistent with the liquid capacity of the column. In both cases the reflux ratio is varied by the temperature control as the feed composition changes, to maintain the required composition of the appropriate product stream.

The selection of the point of temperature control is a matter of some importance and will be discussed in a later article.

Batch distillation

The above remarks have been concerned entirely with continuous columns, but the same principles can be extended to batch distillation columns. The principle difference between the two methods of operation is that in batch distillation a state of continuous steady equilibrium is

(*Concluded on page* 347)

MIXING

Gas-liquid reactions; fermentation processes; continuous mixing; viscous suspensions; functional design of agitators; flow properties of liquids; criteria of mixing efficiency; rate of mixing; draft tubes

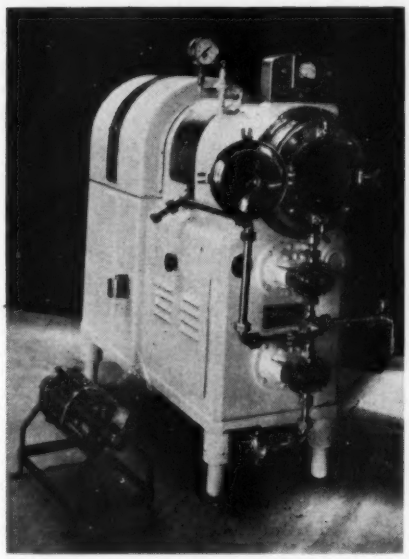
By D. F. Riley, B.Sc., A.C.G.I.

DURING the last 18 months the published literature on mixing has shown a distinct change of emphasis. Now that the influence of the principal factors affecting the power consumption of the standard types of impeller has been quantitatively assessed, more attention is being given to the design of agitation equipment for particular processes and to the task of establishing some sort of fundamental theory of mixing. The papers here reviewed are concerned with the functional design of agitators, the flow properties of liquids, criteria of the degree of mixing, gas absorption, continuous mixing and the agitation of viscous suspensions.

Gas-liquid reactions

In a paper on the role of agitation in process design Green[1] has reviewed the published information on power consumption and the scaling-up of laboratory data to the pilot scale. He considers the effect of agitation on the rate of heterogeneous reactions, which are classified as diffusion controlled, chemically controlled or subject to control by both mechanisms. The reactions most readily influenced by agitation conditions are the diffusion-controlled ones which are of the first order, the reaction velocity varying with the thickness of the Nernst-Brunner molecular layer and with the concentration gradient across it. This class includes many cases of solution of solids and of liquid-liquid extraction where there is no chemical reaction.

Gas-liquid contacting, on the other hand, often involves chemical reaction and the controlling factor may then be diffusion in the gas or liquid film, or chemical reaction in the liquid phase. The work of Hatta on the absorption of carbon dioxide by potassium hydroxide solutions and of van Krevelen on the absorption of carbon dioxide in packed towers is discussed and the more recent theoretical work of Kishinevsky is summarised. An equation deduced by the last-named author is given, for the rate of absorption where a first-order reaction is involved, in terms of the concentration of the liquid, the partial pressure of the gas, and hydrodynamic factors expressing the degree of turbulence. It is concluded that at low turbulence the concentration of the active component in the liquid is more important than the partial pressures of the gas, whereas under conditions of high turbulence the partial pressure of the gas is the decisive factor.



[Creamery Package Mfg. Co. Ltd.

This continuous process mixer relies on scraper blades continually transferring the product from the walls of the cylindrical-type container to the centre, where the reaction-type eccentrically-mounted beater thoroughly whips it and, in some applications, incorporates clean filtered air supplied at any required pressure. The machine is intended to provide continuous production mixing and blending where temperature control of the product is also required.

Green describes some experiments on the oxidation of 400 litres of 0.05% aqueous sodium sulphite solution by air carried out in two nearly identical reactors, each fitted with four longitudinal wall baffles 3 in. wide and a single 12-in.-diam. turbine having six curved blades. The only differences between the reactors were that one was fitted with a draft tube and the agitator speed was 500 r.p.m., whereas the other had no draft tube and the agitator speed was 360 r.p.m. The draft tube increased the interfacial area considerably and gave a rapid turnover of air. It was found, contrary to expectation, that the reactor without the draft tube gave a reaction rate two or three times greater than that given by the other vessel. It was also found that, while the rate of oxygen absorption in the reactor with the draft tube was very low, the rate of a catalysed hydrogenation in the same vessel had been very high. In the ensuing discussion W. S. Norman suggested that the two cases could not safely be compared, since in the hydrogenation a pure gas was being used, whereas in the oxidation at high air rates the unabsorbed nitrogen could 'blanket' the agitator and reduce its efficiency.

Using the same oxygen-sodium sulphite system several different designs of reactor were tested for agitation efficiency. In general the high-speed reactors producing considerable bubble aeration were found to be less satisfactory than those giving little aeration, and it was suggested that this might be due to a reduced effective partial pressure of the gas under conditions of high aeration. Some confirmation of this hypothesis was obtained when the air pressure on a 50-gal. unbaffled reactor with a high-speed propeller agitator was increased by 7 p.s.i. the reaction rate was trebled.

The effect of agitator design and layout can also be seen from Green's results. Of the reactors of 100-l. capacity and greater the baffled turbine gave the best results, while the same turbine with a draft tube gave results little better than were given by an unbaffled propeller. The complex problems which are met when chemical processes are scaled up are discussed generally and it is pointed out that complete similarity is impossible to achieve. However, by appropriate pilot-scale experiments it can be determined whether a reaction is chemically controlled or diffusion controlled and some attempt made to obtain the optimum conditions on the large scale.

Fermentation processes

The literature on fermentation processes and equipment is growing rapidly and much attention has been paid to the effect of agitation and methods of aeration in large-scale fermentors used in antibiotics production, with a view to evolving a technique of scaling-up from pilot-scale data.

The rate of oxygen absorption has usually been measured by means of copper catalysed sodium sulphite solutions, and such factors as the ratio of tank diameter to impeller diameter, the impeller design and speed, the location with respect to cooling coils and air sparger, the degree of baffling and the manner in which the air is introduced have all been considered.

There is some evidence that the ring type of sparger is more effective than the central open air pipe, but more quantitative work is needed to settle the matter. The use of an unbaffled reactor has been reported in which air was introduced over the surface of a deeply vortexing liquid, but apart from its relative simplicity there is reason to believe that it is less efficient as far as the rate of oxygen absorption is concerned.

Maxon and Johnson[2] carried out aeration experiments on the propagation of baker's yeast in a baffled vessel of 3½-l. capacity. Air was introduced by a ring sparger located underneath a propeller agitator and the apparatus was adapted for both batch and continuous fermentations. The rate of oxygen absorption was measured by the standard method for various air rates and agitator speeds and it was found that for a given degree of agitation the rate of absorption (millimoles/l./hr.) was proportional to the 0.4th power of the air rate. Agitator speeds of 500 to 1,680 r.p.m. were used and air rates up to 8,000 ml./min. The growth of the yeast and the production of by-products such as carbon dioxide, ethanol and acetaldehyde was studied and an empirical relationship

$$r = \frac{(3{,}330 - 41.3)c}{y}$$

was developed to allow the oxygen rate required for a given continuous production of yeast to be calculated. In the above equation r = oxygen rate (millimoles/l./hr.); y = per cent. yield of yeast relative to glucose used; and c = production rate (gms. yeast/l./hr.).

The scale-up of penicillin fermentation equipment from a 5 gal. through a 2,000 gal. to a 24,000 gal. commercial fermentor is described by Wegrich and Shurter.[3] The 2,000-gal. unit had four longitudinal baffles located 1½ in. from the wall and the agitation was provided by 3 in. to 28 in. diam. flat-bladed turbines. The agitator speed varied from 80 to 150 r.p.m. and the power input from 0.3 to 2.5 h.p./100 gal. Filtered air at 24 p.s.i.g. pressure was was metered to the fermentor, the rate varying up to 150 cu.ft./min. Since the power consumption alters with the tendency to foam formation and with the changes of viscosity and density during the course of the fermentation a recording wattmeter was used to enable an average power consumption to be estimated.

Scaling-up was calculated on a basis of equal back pressures in the model and full-scale fermentors and on equal superficial air velocities. The effect of agitation on penicillin production was studied and it was found, using air at 27 p.s.i.a. and at 2.1 ft./min., that the production rate in units/ml. increased linearly with time, provided the power input exceeded about 0.5 h.p./100 gal. At 0.32 h.p., however, the rate fell off during the last third of the fermentation time. The large production vessel was not in all respects geometrically similar to the 2,000-gal. vessel and the scaling-up was based not on dynamic similarity but on equality of power input, the figure used being 0.7 h.p./100 gal. at an air velocity of 2.1 ft./min.

[*Kestner Evaporator & Engineering Co. Ltd.*

In the new Kestner twin shaft (patents pending) stirrer, two shafts are employed, both working in parallel driven by a common driving motor through a suitable gear box. Each shaft carries one or more propellers and rotates in the same direction, *i.e.*, clockwise, when viewed from above. This arrangement of the shafts and propellers is claimed to have three main advantages. Firstly, the number of zones of agitation is increased, secondly, each zone operates in opposition to the next, so causing a more efficient and overall degree of agitation, and, thirdly, mass rotation of the liquor, normally experienced when only one shaft is used, is eliminated. The illustration shows one of these stirrers mounted on the rim of a jacketed pan by means of an adjustable bracket and simple hand-operated screw clamps. This method of mounting enables the angle of the shaft to be adjusted and the complete unit easily removed for use on other vessels. The unit has a total weight of approximately 80 lb. Each shaft is held by a quick action collet chuck which enables the length to be adjusted to a maximum of 5 ft. 6 in. and the position of the propellers can be adjusted at will. The stirrer illustrated is fitted with a ½ h.p. totally enclosed motor and gear box giving the output shafts the normal speed of 440 r.p.m. This machine is stated to give intense agitation in up to 100 gal. of a fluid having a viscosity of 8 poises and a specific gravity of 1.8, and mild agitation in up to 200 gal.

Continuous mixing

In a paper by Weber[4] on the design of continuous reactors the relative advantages of batch and continuous systems are discussed. Continuous operation has the obvious advantages of reducing labour costs, eliminating time required for charging and heat transfer, and improving uniformity of product. It suffers, however, from the defect of an inherent decreased overall reaction rate, especially noticeable in organic systems requiring long reaction times, although this may be outweighed in practice by the advantages enumerated. It is pointed out that the production rate from a continuous reactor is very sensitive to the degree of completion of reaction required. If a high degree of completion (90 to 95%) is not essential, the continuous unit usually has a greater production capacity than the batch plant. When, however, a completion of (say) 99% is necessary, a batch reactor will have the advantage over a continuous plant, unless the latter employs several reactors connected in series.

Weber has presented a graphical method for determining the number of reactors in series required to achieve a given composition of effluent when the average holding time is specified. The procedure is based on reaction velocity data, which can be obtained from an experimental batch plant, and on the equation

$$\theta = (f_n - f_{n-1})\frac{c}{K_n}$$

Where θ = average holding time; f_n = fractional degree of completion in the nth reactor; K_n = reaction velocity in the nth reactor; and c = initial reactant concentration.

The problem of short circuiting in continuous reactors is discussed and a general equation is developed which enables the theoretical degree of short circuiting to be calculated for a reactor having some positive means (such as a draft tube) of directing the feed through the zone of intense agitation. The proportion of feed leaving a continuous reactor system is given by the expression

$$\frac{(n-1)!}{(m-1)!\,(n-m)!}\left(\frac{F}{Q}\right)^m\left(1-\frac{F}{Q}\right)^{n-m}$$

in which n = the number of passes of reactants in the vessels; m = the number of reactors in series; F = volumetric throughput rate; and Q = recirculation rate of the agitator.

Weber stress the importance of the ratio $\frac{F}{Q}$ which can be made the basis of scaling-up from a continuous pilot reactor. In scaling-up from a batch reactor to a continuous one this ratio cannot be used and the relevant criterion is the ratio $\frac{Q}{V}$ where V is the reaction volume. Unfortunately,

no experimental data are given in support of the theories advanced. In the subsequent discussion R. B. MacMullin suggested a new method for determining the relative efficiency of different types and speeds of agitator, namely by studying their effect on the reaction rate of a system of known kinetics.

Viscous suspensions

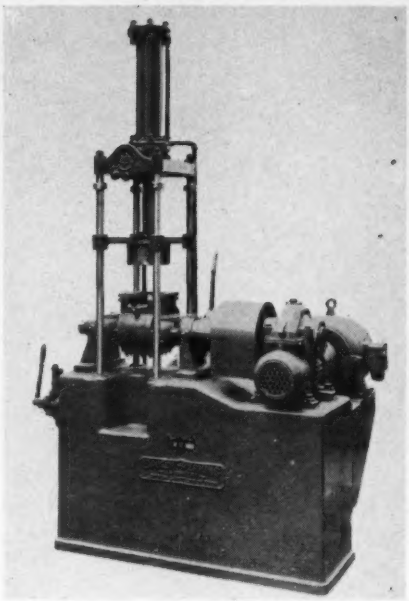
Although the majority of agitators are applied to the task of creating turbulence in liquids of low viscosity, there is a significant number of occasions (*e.g.* in the food and plastics industries) when materials of high viscosity have to be handled and any flow created is likely to be streamline. In this field new ground has been broken by Hirsekorn and Miller,[5] who have carried out extensive experimental work to evaluate the factors involved in the agitation of suspensions of solids in viscous liquids. They used several unbaffled cylindrical glass vessels from 5.6 in. to 17.6 in. diam. and simple two-bladed flat paddles 1.44 in. to 15.75 in. diam. Corn syrup solutions were made, having viscosities ranging from 3,800 to 92,000 cp., the viscosity in each experiment being adjusted so as to keep the Reynolds number from exceeding 10. Several closely sized fractions of sand particles were prepared with settling velocities of 1.8 to 3.7 10^{-4} ft./sec. and power measurements were made by means of a dynamometer in suspensions having a concentration of 2% by weight.

In order to obtain a single curve for all geometrically similar impellers over the range of Reynolds numbers studied, Hirsekorn and Miller eliminated tank diameter and liquid depth from the original power function used by White and his co-workers. They correlated their results in terms of the relation

$$N_p.\mathrm{Re}.\left(\frac{D}{W}\right)^{0.5} = \phi\,(\mathrm{Re})$$

where N_p is the usual power function $\frac{P}{\rho N^3 D^5}$, D is the paddle diameter and W the paddle width. Good agreement is claimed between these results and those obtained by O'Connell and Mack for paddles in baffled tanks in the viscous region, confirming that under streamline conditions wall baffles have almost no effect. The clearance of the paddle from the bottom of the tank had, within practical limits, negligible effect on the power consumption. The viscosity of the liquid phase has two opposing effects on the suspensions of solids; it hinders the initial formation of the suspension by restricting the flow pattern, but on the other hand it assists the agitator to maintain the suspension, once formed, by reducing the settling velocity of the particles.

In a series of experiments with geometrically similar models covering a scale range of 3:1, Hirsekorn and Miller increased the speed of each agitator in steps in order to determine the minimum speed for complete suspension and they found that this always occurred at about the same power input per unit volume of slurry, although the viscosity varied several-fold over the range of sizes. The height of solids in the suspension was 60 to 80% of the liquid depth and it is suggested that the solids-free supernatant layer is a result of the geometry and fluid dynamics of the system rather than due to any inherent inability of the agitator to project the particles to the surface of the liquid. The most effective paddles were those with a ratio of tank diameter to paddle length of 1.1 to 1.7 and the power requirement was of the order of 0.15 to 0.20 h.p./100 gal.



[Baker Perkins Ltd.

A heavy-duty laboratory mixer having a working capacity of .5 gal. and with a 4-h.p. drive. The trough is jacketed and the blades cored for temperature control and a pneumatically operated ram is provided to confine the material under treatment under the action of the blades. This small unit can be employed for the mastication of crepe rubber, the production of aqueous rubber dispersions, the dispersion of finely divided pigments in natural and synthetic mediums and a variety of other tasks.

Functional design of agitators

Two papers by Rushton and Oldshue[6] provide a comprehensive survey of present theory and practice in the field of liquid mixing. Emphasis is placed on the fluid mechanical approach to the problems of mass transfer and turbulence involved. The flow patterns obtained in the commoner arrangements of vessel, with and without baffles, and impeller, located centrally or off-centre are described. The elements of the theory of turbulence are briefly reviewed, particular mention being made of the concepts of eddy viscosity and 'mixing length.' A useful start has been made in attacking the problem of determining the rate of mixing induced by agitator impellers, by considering the amount of entrainment induced by a submerged jet. A quantity of water Q issuing from a submerged jet of diameter d has been found (Folsom and Ferguson, 1949) to entrain water at a rate equal to $(0.23\frac{x}{d} - 1)Q$ where x is the distance along the axis of the jet. The power characteristics of mixing impellers are considered and values of the proportionality constant k in the general power equation $\frac{Pg}{\rho N^3 D^5} = K\mathrm{Re}^n$ are given for all the common types of impeller, for conditions of streamline flow ($n = -1$) and fully baffled turbulent flow ($n = 0$) assuming certain standard geometrical ratios. Experimental work already done on the flow from three-bladed marine propellers and now in progress on the flow from radial flow turbines is described, a photographic technique being adopted using traces made by moving droplets of an immiscible liquid to reveal the flow patterns. Analysis of such flow patterns gives insight into the ways in which turbulence is generated in an agitated liquid. It appears that, although some of this turbulence is initiated at the boundary walls of the vessel and at baffles, most of it is due to the contact between the high-velocity stream leaving the impeller and the lower-velocity stream in the surrounding liquid.

In order to design an agitator rationally to perform some specified duty such as blending liquids, suspending solids, improving heat transfer or contacting a gas and a liquid, it is necessary not only to determine the size, layout, speed and power of the impeller but also to relate the flow and head to some rate coefficient of mass transfer or heat transfer. For example, the general equation relating agitation and heat transfer is

$$\phi\,(\mathrm{Nu}, \mathrm{Pr}, r_1, r_2 \ldots\ldots) = K\mathrm{Re}^x$$

where Nu = the Nusselt number, Pr = the Prandtl number, $r_1, r_2 \ldots\ldots$ are other geometric and dynamic ratios relevant to the particular conditions and Re = the Reynolds number. For mass transfer a similar equation holds, but with Nu replaced by the analogous group $\left(\frac{kL}{D}\right)$ and Pr replaced by the Schmidt number $\left(\mathrm{Sc} = \frac{\mu}{\rho D}\right)$ where D is the diffusivity and k is the mass transfer coefficient. The exponent x is known as the 'mixing slope' and it is shown that, when the value of x is known, the ratio of power per unit volume on the full scale to that of a geometrically similar pilot scale may be calculated for any given scale ratio and, further, that only when $x = 0.75$ is the power per unit volume the same regardless of the scale factor. The principal data on agitation

and heat transfer are summarised, the values of the mixing slope varying from 0.62 to 0.90.

The specific agitation requirements for such operations as heat transfer, suspension of solids and contacting of immiscible liquids are discussed, with particular reference to the ratio of flow to head necessary. It is shown that the effect of speed on the flow from and head developed by an impeller at constant power input may be calculated by suitable rearrangement of the relevant equations, and that under these conditions the Q/H ratio changes rapidly with speed. In general it is found that high ratios of flow to head are required for blending liquids and suspending solids, while low values are better suited to gas liquid contacting and the mixing of immiscible liquids. A final section of these papers is devoted to a brief discussion of some mechanical aspects of liquid mixer design.

Flow properties of liquids

A recent article by Quillen[7] provides a general survey of the equipment normally used for mixing liquids, pastes and solids, and includes some account of the theory of mixing. The design of agitation equipment should always start from a consideration of the flow properties of the materials being handled, and in this connection Quillen rightly emphasises the importance of a rheological approach. He divides the non-Newtonian properties of materials into those which are time-dependent and those which are independent of time. Certain suspensions of approximately equidimensional solids, such as sand in water, and drilling muds, have an interlocking structure and show what is known as Bingham plasticity (see Fig. 1). Flow does not start until the shear stress exceeds a certain minimum (the yield stress) and the rate of shear thereafter increases linearly with the shear stress, as it does with Newtonian liquids.

Pseudoplasticity is the name given to the type of flow in which, as the shear stress increases, the increment of shear stress necessary to produce a given increase in the rate of shear decreases rapidly. It is shown by some suspensions or solutions of long thin particles such as organic polymers, cellulose acetate, cooked starch pastes, paper pulp, soap and detergents. The shape of the characteristic flow curve is thought to be due to the progressive alignment of the particles in the direction of flow. A type of flow, known as dilatancy, is shown by uncooked starch suspensions and by concentrated deflocculated pigment dispersions. This is characterised by behaviour which is opposite to pseudoplasticity in that, as the shear stress increases, the increment of shear stress necessary to produce a given increase in the rate of shear increases rapidly.

The two types of flow behaviour which are dependent on time are known as thixotropy and rheopexy (see Fig. 2). A liquid

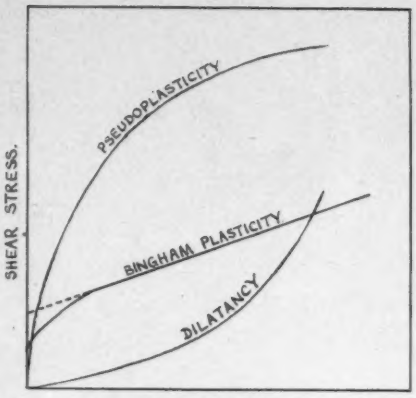


Fig. 1.

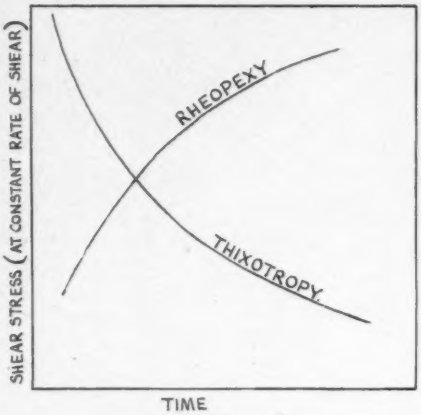


Fig. 2.

is said to show thixotropic behaviour when the shear stress required to maintain a given constant rate of shear decreases with time, and rheopectic behaviour when it increases with time. The internal structure of a thixotropic liquid is broken down under stress, but the degree of alignment of the particles depends on time as well as on the rate of shear. If shear stress is plotted against rate of shear for a thixotropic liquid two curves are obtained depending on whether the rate is increasing or decreasing, and the area of the hysteresis loop is a measure of the amount of thixotropy. This type of flow is shown by solutions of some organic polymers, by paints and printing inks, and by food products such as mayonnaise, while rheopexy is exhibited by suspensions of gypsum and bentonite, and by vanadium pentoxide sols.

Criteria of the degree of mixing

In an interesting and important paper Danckwerts[8] an attempt is made to establish some fundamental criteria by means of which one can decide how well a number of components have been mixed. The ' scale of scrutiny ' is defined as the minimum size of the regions of segregation in a mixture which would cause it to be regarded as imperfectly mixed for a specified purpose. It may vary from the order of magnitude of the mean free path of the molecules in a gas mixture to a dimension readily visible to the naked eye in a coarse-grained mixture of solid particles. A coarse-grained mixture is one where the scale of scrutiny is not large enough to include many particles. Such a mixture may be fully ' randomised,' but each component will not be perfectly dispersed with respect to the others. There will be local fluctuations of concentration which will be significant because the allowable scale of scrutiny is not large enough. Applying statistical theory to such cases it is possible to decide how well the components are mixed by observing the variations between samples. A fine-grained mixture, on the other hand, is one for which the scale of scrutiny is large enough to include very many particles of each component. The fluctuations in composition which must exist in such a mixture when fully ' randomised ' are insignificant with respect to the allowable scale of scrutiny. Hence for practical purposes such mixtures are regarded as having uniform composition.

Danckwerts has studied the measurement of goodness of mixing of fine-grained mixtures and has found that, in order to specify it fully, two variables are required known as the ' intensity of segregation ' and the ' scale of segregation.' The scale of segregation, v, is a measure of the size of the regions of segregation in an imperfect mixture, while the intensity of segregation, I, is a measure of the departure of the composition from the mean value. If a series of samples of given size drawn from a mixture are required to show not more than a certain specified variation in composition, it is the product Iv which must be reduced to the necessary value by mixing. The intensity and scale of segregation are related to the time and other variables of agitation by the following equations

$$I = f_1\left(\mathrm{Re},\ \mathrm{Fr},\ \mathrm{Sc},\ \frac{ut}{L}\right)$$

$$\frac{v}{L^3} = f_2\left(\mathrm{Re},\ \mathrm{Fr},\ \frac{ut}{L}\right)$$

where Re = Reynolds number, Fr = Froude number, Sc = Schmidt number, u = a characteristic velocity, L = a characteristic linear dimension and t = time required to achieve a given scale and intensity of segregation. The function of a mixer is to reduce the scale of segregation as rapidly as possible, and the intensity of segregation will be reduced by molecular diffusion. In the mixing of heavy pastes the scale of segregation is progressively reduced by cycles of superposition and deformation. In such processes diffusion plays little or no part, but the interfacial area between the components continually increases.

Rate of mixing

A comparative study of the rate of mixing in agitated tanks has been made by Kramers, Baars and Knoll.[9] The speed of distribution of a small amount of potassium

[*Sturtevant Engineering Co. Ltd.*

A batch blender capable of mixing 3 tons of glass batch at one time. The machine comprises a steel drum of large diameter and narrow width. A series of flights or buckets is mounted on the internal circumference of the drum and these flights gently cascade the material over and over, thus giving a very intimate mix. One end of the drum is carried in a self-aligning ball-bearing on a stub shaft rigidly fixed to a substantial bracket mounted on the base frame, whilst the other end of the drum is supported in a trunnion bearing to which is fitted a dust-tight packed gland. At this end also is a valve box through which the materials are introduced and discharged, and a swinging chute is placed inside the drum which is linked to the valve-operating levers. During mixing, the chute hangs freely in the drum allowing the materials to further cascade and improve the blending action, but when the machine is being discharged the chute automatically causes the material to flow out through the bottom of the discharge box. The mixing drum is rotated by means of a chain drive.

chloride solution added to water was measured under various conditions of agitation. Potassium chloride solution was used because its electrical conductivity is known to be directly proportional to the concentration over the range 1 to 20 mg./cc. The concentration difference between two fixed points was measured by means of conductivity cells connected to a Wheatstone bridge. The difference in conductivity relative to the average in the vessel was recorded so that the time could be read from the charts corresponding to 'complete' mixing. The criterion of complete mixing in these experiments was arbitrarily selected as a concentration difference equal to 0.1% of the mean concentration.

Two vessels were used, of 12½ in. and 25 in. diameter, and the liquid depth was maintained equal to the vessel diameter. Three impellers were tested—a three-bladed propeller, a turbine with six flat radial blades and a turbine with six flat blades swept back at an angle of 30°. Four longitudinal baffles at the tank wall and set one width away from the tank wall were compared with one another and with a cruciform baffle on the bottom of the tank. Experiments with the propeller in the unbaffled tanks confirmed earlier results, in that the time of mixing was reduced by placing the agitator shaft off-centre. When the propeller shaft was inclined 15 to 30° to the vertical, however, more rapid mixing was obtained with the shaft directed across a diameter of the vessel.

Wall baffles were found to be more effective than those set away from the wall when the propeller was used, and the cruciform baffle was effective only if the vertical clearance of the propeller from the bottom of the tank was small. When the turbines were tested the baffles at the tank wall were not so effective as those away from the wall. The turbines were able to give as rapid mixing as the propeller but at greater power consumption under the same conditions. When the larger of the tanks was used the torque on the agitator shaft was measured and the power consumption, in the range $Re = 4.10^4$ to 10^5, was found to be independent of the Reynolds number, as would be expected for fully turbulent flow in a baffled tank. For geometrically similar systems, using the same liquid and with agitators running at the same peripheral speed, the total energy used per unit volume was shown to be the same, the time of mixing being proportional to the scale factor.

Draft tubes

The various uses of draft tubes in agitated vessels form the subject of a paper by Weber.[10] The use of a helical screw in a draft tube is time honoured in the soap industry, being the essential mechanism of the soap crutcher. It can only be used for pasty materials, as it depends on the high viscosity to reduce slip between the screw and the inner surface of the draft tube. Weber also describes the application of draft tubes to continuous mixers in order to prevent short circuiting, and to gas-liquid contacting in unbaffled vessels for the purpose of establishing vertical flow and creating a vortex. A draft tube may also be used to assist an agitator to suck in solids floating on the surface of a liquid, and in the mechanical dispersions of solids in liquids by forcing the suspension through a perforated plate closing the bottom of the tube.

REFERENCES

[1] S. J. Green, *Trans. I. Chem. E.*, 1953, **31**, 327.
[2] W. D. Maxon and M. J. Johnson, *Ind. Eng. Chem.*, 1953, **45**, 2554.
[3] O. G. Wegrich and R. A. Shurter, *Ibid.*, 1953, **45**, 1153.
[4] A. P. Weber, *Chem. Eng. Prog.*, 1953, **49**, 26.
[5] F. S. Hirsekorn and S. A. Miller, *Ibid.*, 1953, **49**, 459.
[6] J. H. Rushton and J. Y. Oldshue, *Ibid.*, 1953, **49**, 161, 267.
[7] C. S. Quillen, *Chem. Eng.*, 1954, **61** (6), 177.
[8] P. V. Danckwerts, *Research*, 1953, **6**, 355.
[9] H. Kramers, G. M. Baars and W. H. Knoll, *Chem. Eng. Sci.*, 1953, **2**, 35.
[10] A. P. Weber, *Chem. Eng.*, 1953, **60** (10), 183.

Automatic Control of Distillation Processes

(*Concluded from page* 342)

never attained. Since there is no feed to the still—the feed stock being normally charged directly into the still base—the composition of the liquid in the reboiler is continually changing as the lighter components are stripped out. Thus the temperature and composition gradients in the column will change continuously throughout the batch. As a result the reflux ratio must be continuously increased, from a minimum at the start of distillation to a condition of total reflux at the end when all the overhead product has been removed.

The conditions are rather similar to a continuous column with a feed of changing composition, and the control is therefore based on the overheads composition, *via* the temperature at a suitable point, by regulation of the reflux return (or product take-off). It is also possible to arrange by suitable programme and time-cycle units that, at the end of the distillation, the column is shut down or the product receiver is changed and a higher boiling fraction removed.

The heat input to a batch still is usually held constant by flow control, but can be regulated by the column pressure differential to give a maximum vapour velocity. Control of the remaining variables, the column static pressure and the condenser cooling are exactly the same as in continuous distillation.

In a later article the control of the individual process streams will be considered in rather more detail, and examples of typical applications will be considered.

Refractory bricks are described and illustrated in three new leaflets issued by the Morgan Crucible Co. Ltd. The M.I.23 can be used as a direct furnace lining (*i.e.* as a hot-face insulator) up to 1,260°C. (2,300°F.) where furnace conditions are clean and, where furnace temperatures are higher, as a backing insulation to a lining of denser refractory or of another hot-face refractory such as M.I. 28.

The M.I. 28 is a hot-face low-heat-storage insulating refractory which can be used at furnace (or interface) temperatures up to 2,800°F. (1,538°C.). At these temperatures it has a lower conductivity than any other type of refractory, and therefore provides a greater reduction in the convection and radiation losses from the outside of the furnace.

The M.R. 1 is a high-quality firebrick which is claimed to show a high degree of stability at operating temperatures up to 1,600°C. and an after-contraction figure for 2 hr. at 1,550°C. of less than 1% (the usual temperature for measuring after-contraction is 1,410°C.). It is further stated that the bricks are close-textured and strong, and their resistance to abrasion is high.

METALLURGY

New metallurgical laboratories; boron steels; machining of metals; corrosion; metal finishing; instruments; titanium developments; molybdenum; zirconium; aluminium

By H. A. Holden, M.Sc., A.R.C.S., D.I.C., A.I.M.

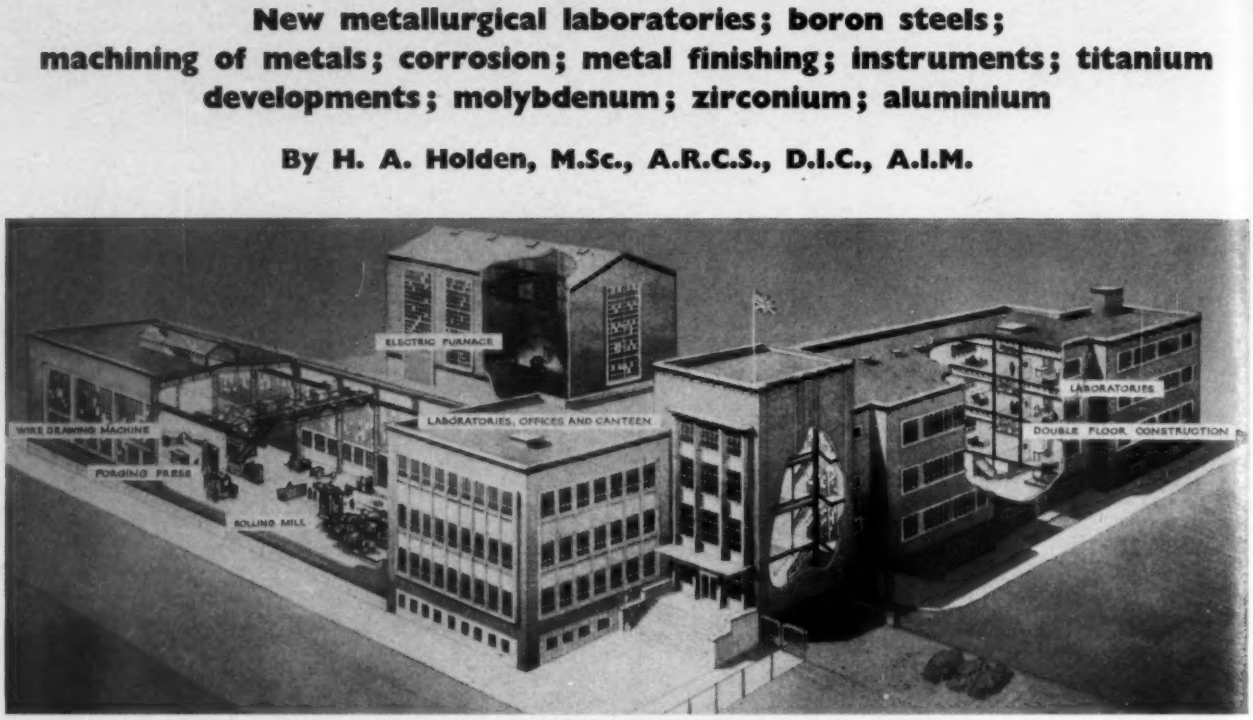


Fig. 1. New laboratories of the British Iron and Steel Research Association at Sheffield. *J. Iron & Steel Institute*

FERROUS

FACILITIES available in this country for metallurgical research and development, with special reference to the steel industry, were greatly increased when H.R.H. the Duke of Edinburgh opened the new Sheffield laboratories of the British Iron and Steel Research Association on November 19, 1953.[1] These laboratories (Fig. 1) comprise a main laboratory and office block, a metalworking shop and a melting shop. A most interesting feature of the main block is that all the internal walls are easily removable to allow new layouts to be built, the main services being carried in four vertical brick-built shafts from the basement to an easily accessible system of horizontal ducts running in false ceilings to the corridors and rooms. As a result, offices can be converted into laboratories and *vice versa* without any structural alterations.

The metallurgical laboratories are equipped for heat treatment; metallurgical examination; mechanical, magnetic and electric testing; *x*-ray examinations; and chemical, spectrographic and vacuum-fusion analysis. The single-storey metal-working laboratory contains a strip rolling mill, a forging press and a wire-drawing machine, and the steel-making laboratory houses a 10-cwt. electric arc furnace and the furnace transformer. A central design, machine shop and maintenance section deals with the construction of apparatus and the preparation of specimens required for the three research divisions: steelmaking, mechanical working and metallurgy.

A description has also been published of the facilities available and investigations carried out at the Sketty Hall, Swansea, laboratories of B.I.S.R.A.[2] Here the work of the research team, which numbers about 30, is divided into four sections: electrochemistry, metallurgy, physics and chemistry, and is concerned with the application of all metallic and other inorganic coatings to steel, and with steel surfaces in general. The electrodeposition of iron-zinc alloys is receiving considerable attention and alloys have been deposited with zinc contents varying from 3 to 90%. The 6% alloy is nearly as reflective as a perfect mirror (Fig. 2), has good adhesion to the steel sheet on which it has a smoothing effect, can be deposited at a rate of 0.0001 in./min., has a hardness of 560 D.P.N., but although it is tarnish-resistant indoors, it does not resist corrosion sufficiently well to be used outdoors. Other projects include the development of a simple continuous hot-dip method of coating steel strip with aluminium, the recovery of zinc from galvanisers' dross and the recovery of sulphuric acid from waste pickle liquor.

Boron steels

Boron steels continue to be the subject of intensive research and development. In fact, they were considered to be so important that a special meeting on boron in

Table I. Typical Applications of Boron Steels (Knowlton[3])

Part	*Size*	*Brinell hardness*	*Quench*	*Original steel specification*	*Alternative boron steel*
Shafts	>3 in.	Up to 450	Oil	4340	86B45
Flat springs	1–2 in. thick	Up to 450	Oil	9262	51B60
Coil springs	1–3 in. dia.	Up to 450		8660	51B60
Flat and coil springs	<1 in. dia.			9260	50B60
				5152	80B60
Shafts, forgings, heat-treated parts	<2 in. dia.	Up to 450	Oil	4140	81B40
	3 in. dia.		Water	8640	80B40
	>3 in. dia.		Water		50B40
Bolts	<9/16 in.		Oil	4130	
	1–1¼ in.		Water	4035, 3130	14B35
Bolts requiring difficult cold heading			Oil	4037	40B37

steel arranged by the Iron and Steel Institute at the suggestion of the Ministry of Supply, acting in conjunction with the Organisation for European Economic Co-operation, was held in London on April 29, 1953. At this, papers on 'The Manufacture and Use of Boron Steels in the United States,' by L. J. Rohl (United States Steel Corporation) and 'American Applications of Boron and other Low-alloy Steels,' by H. B. Knowlton (International Harvester Co.) were presented and discussed.[3] The only British paper, 'Effects of Boron on the Mechanical Properties of Low-alloy Steels,' by R. Wilcock (Samuel Fox & Co. Ltd.) was considered in some detail in the previous review in this journal.[4]

The marked increase in production of boron steels in the U.S.A. (see Fig. 3) is reported by Rohl, who states that in 1951 this use of boron steels was responsible directly or indirectly for the saving of about 12 million lb. of nickel. According to Knowlton, about 8 to 10% of the tonnage of American low-alloy constructural steel has been converted to lower-alloy steels containing boron, with satisfactory results. Further, his company has used about 80,000 (short) tons of chromium-boron steels per year in the manufacture of trucks and tractors and no failures attributable to the steel have been reported. Typical applications of boron steels are shown in Table 1. In addition, clutch plates formerly specified as 0.55% Ni, 0.50% Cr, 0.20% Mn steel are now made of 0.30% Ni, 0.40% Cr and 0.12% Mo boron-treated steel, and have proved most satisfactory in service.[5] Other general reviews of the development, present position and applications of boron steels have been published by J. F. Sewell[6] and G. Delbart.[7]

On the rather more academic side, R. N. Imhold and J. W. Poynter[8] have compared the physical properties—with special reference to properties of interest to the aircraft industry—of three American boron

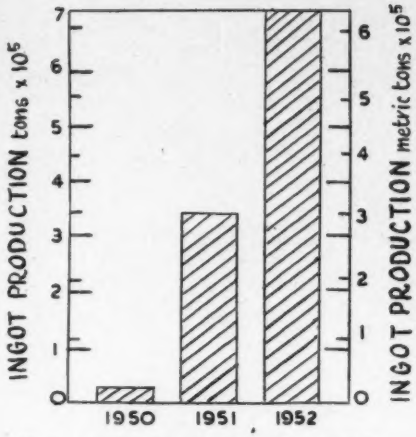


Fig. 3. Approximate production of boron steels in the U.S.A.

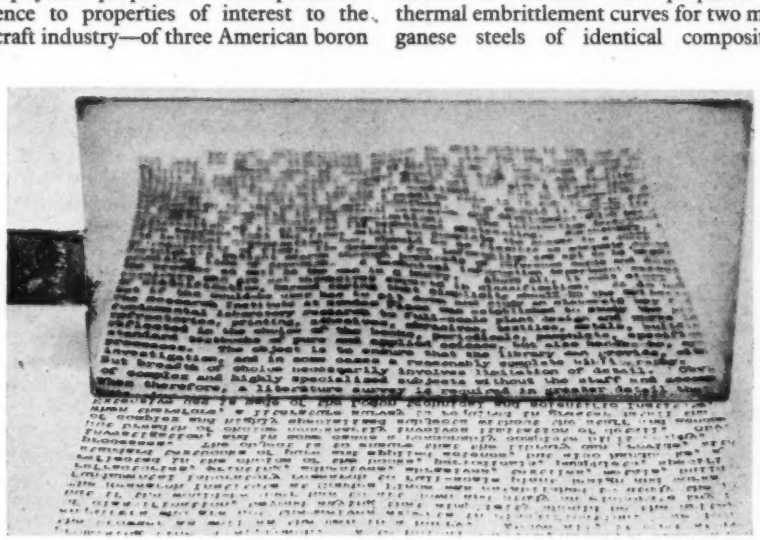
steels containing 0.3 to 0.43% C with those of the standard steels which they replace. N. Zlatin, J. F. Kahles and W. F. Friedlander[9] have described tests at Waterton arsenal on six standard alloy steels and seven equivalent boron steels, in which both surface finish and test life were compared. They found that the boron steels possessed somewhat better machining characteristics, the improvement being more pronounced with high-speed steel than carbide tools. Machinability has also been discussed by G. D. Rahrer,[10] who also found that with the steels he examined boron had no adverse effect on weldability. Quantitative comparisons of the effect of boron on the hardenability of carbonitrided and carbonised cases[11] and on cementation and heat-treatable steels[12] have been reported, and the effect of making the boron additions as either a simple 12% boron alloy such as ferroboron or as a more complex alloy such as Grainal (Bo, Si, Ti, Al, Mn, Zr) studied.[13, 14] A. E. Powers and R. G. Carlson[15] have prepared isothermal embrittlement curves for two manganese steels of identical composition except that one contained 0.0034% Bo. Susceptibility to reversible temper embrittlement at 850 to 1,050°F. was increased by the boron addition. Finally, working hypotheses for the mechanism of boron hardenability have been postulated.[16, 17]

Machining

Although of considerable practical significance, the machinability of metals and machining operations have often received too little attention from both metallurgists and engineers. During recent years, however, the machining of hard metals has been the subject of considerable development, with particular reference to 'electric spark machining' techniques. In the method developed by Sparcatron Ltd., Gloucester, the machine applies a continuous spark; the work (positive) and electrode (negative) being immersed in a hydrocarbon liquid dielectric, which in addition to its cooling action flows so as to remove the distintegrated particles. According to D. W. Rudorff,[18] this process is particularly valuable for cutting cemented carbides and highly alloyed heat-resistant metals. He suggests that the material removing action of the electric spark discharge is due to its high temperature, spark discharge taking place so quickly that the surrounding metal remains cool and the material at the spark seat is instantly vaporised. In consequence, the resultant surface finish consists of a multitude of very small pits, which are useful in some instances for the retention of lubricants. F. F. Cherepanov[19] has described Russian work using a somewhat similar principle for the electric spark sharpening and shaping of hard alloy-tipped cutting tools. In this device, the lathe spindle is provided with a chuck holding the cutting disc (negative). The machined part is given a positive charge and is, of course, insulated from the lathe stand. Copper was found to be superior to iron or steel as the cutting disc, highest productivity being obtained at 50 v. and 25 to 30 amp., using machine oil as the liquid. The whole subject of electrospark machining has been reviewed by C. R. Alden,[20] who gives some details of the 'Method X' type of machine.

M. E. Marchant, H. Ernst and E. J. Krabacker[21] have developed a rapid method of measuring cutting-tool life using radioactive isotopes as tracers. Machining is performed using a tool which has been made radioactive by neutron irradiation, collecting the resultant chips and measuring their radioactivity. B. Colding and L. G. Erwall[22] have described similar tests carried out in Sweden using W^{187}.

Carbon dioxide is now being used as a coolant for metal-working tools and this development is well past the laboratory stages.[23] The best field of application is in dry turning, where increased speeds and extended tool life are claimed. Details have been released of the 'CeDeCut' process marketed by the Carbon Dioxide Co.,



J. Iron & Steel Institute

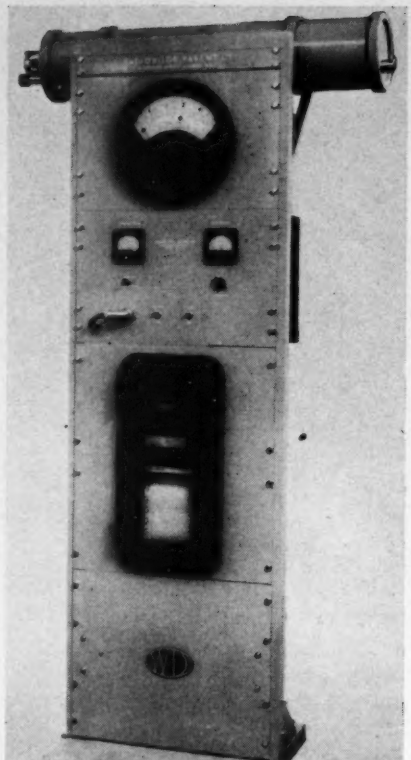
Fig. 2. Reflection by an iron-zinc alloy coating (photo negative was reversed).

a division of The Distillers Co. Ltd.,[24] which is claimed to intensify the cooling action of the carbon dioxide and eliminate the waste involved in needlessly cooling the chip. Turning speeds of 300 ft./min. for Nimonic 75 and 950 ft./min. for high-tensile steel were obtained during experimental trials.

Corrosion

Fretting corrosion, although recognised for many years as no true corrosion effect, is coming into the news. It is a surface deterioration occurring when closely fitting metal (or non-metal) surfaces move only minutely with respect to one another as, for example, when they are subject to vibration. A comprehensive review of the factors which influence, or are thought to influence, fretting has been published by W. E. Campbell.[25] Initiated by mechanical action, chemical action only follows when reaction with the environment is possible and, according to Campbell, it can be reduced but not eliminated by correct lubrication. Many mechanisms have been suggested, amongst them the repeated 'making and breaking' of cold welded surfaces accompanied by accelerated oxidation. A. H. Allen[26] has described experiments in which fretting was induced in specimens under microscopic observation, but the most thorough investigation of fretting phenomena is undoubtedly that carried out in this country by K. H. R. Wright.[27] Two lapped carbon-steel surfaces were loaded against each other, subjected to slight oscillatory movement and quantitative measurements of the fretting damage made. The degree of corrosion was correlated with the number of oscillations, load and particularly with the atmospheric humidity. Lubricated phosphated surfaces were found to be especially valuable in reducing or inhibiting fretting corrosion. According to D. Chambaud,[28] shot peening, cyaniding or carbonising increase the resistance to fretting, and lubricants containing an aluminium-base soap have proved superior to normal-type lubricants. Nickel plating has considerably reduced the ravages of fretting corrosion on the bearings of rolling mills. In view of its increasing importance to metallurgists, chemical and mechanical engineers, fretting corrosion is clearly destined to receive much closer and more detailed study in the next few years.

In the more orthodox corrosion field, J. C. Hudson and J. F. Stanners[29] have published the results of sea-immersion (six years) and atmospheric-exposure (12 years) tests on the protection of iron and steel by metallic and non-metallic coatings. This paper should be read by all who are in any way connected with the protection of steel from atmospheric or marine corrosion. Their work has confirmed that the protective value of aluminium, cadmium and zinc coatings on steel is roughly proportional to the coating thickness. They conclude that, in an industrial atmosphere such as Sheffield, flat steel surfaces can be protected against rusting for 10 years by a coating of aluminium ($\frac{3}{4}$ oz./sq.ft.), zinc (3 oz./sq.ft.) or lead (3 oz./sq.ft.). These correspond to coating thicknesses of 3 mils for aluminium and lead and 5 mils for zinc. In industrial atmospheres, aluminium coatings protect steel against general corrosion rather better than zinc coatings of equal thickness, which in turn are slightly better than cadmium coatings of an equal thickness. Further, lead coatings are less serviceable in rural and marine atmospheres than in an industrial atmosphere where a protective film of sulphate is produced. Zinc is the best of the metals tested for protecting steel immersed in the sea. A coating weight of 3 oz./sq.ft. will prevent serious rusting for about six years.



[Woodall-Duckham Construction Co. Ltd.

The control panel, complete with sampling chamber, of the B.I.S.R.A. blast furnace gas dust density measuring equipment. The sampling chamber fitted at the top of the panel is fed with a sample of gas extracted from the main ducting which has passed through a preheating chamber, thus raising it above dew point. The indicating instrument is a 7-in.-diam. meter which incorporates alarm contacts to energise a warning bell in the event of a high dust concentration passing through the sampling chamber. The control unit, fitted with setting-up meters, is mounted below, having a multi-point plug and socket for connection to the sampling chamber. A continuous record of the level of dust concentration is provided by means of the flush-mounted 3-in.-wide chart recorder.

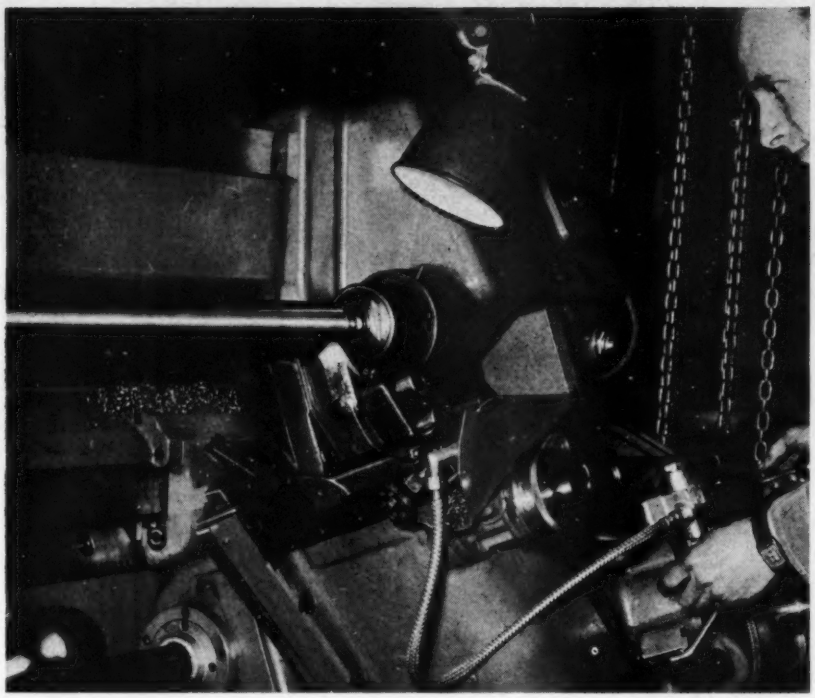
Metal finishing

There is still considerable interest in America in the so-called electroless nickel-plating processes[30] by which nickel coatings (90 to 95% Ni) are deposited from solution as a result of chemical reaction instead of electrodeposition. The chief advantages are claimed to be that the coatings are applicable to intricate parts which cannot be successfully electroplated, and afford better corrosion protection for a given thickness. According to E. L. Gostin,[31] the hardness, wear and corrosion-resisting properties of the films are improved by low-temperature heat treatment. Production plants have been described by H. J. West[32] and T. A. Dickinson.[33] In the first of these, two 9-gal. (U.S.) pots 'plate' about 15,000 parts in 8 hr. The solution, which is operated at 200°F., contains nickel chloride, sodium hypophosphite and sodium citrate. The other, which treats ferrous and cuprous materials, is located at the Cheamplate Co., Los Angeles, the solution constituents being nickel chloride, sodium hypophosphite, sodium hydroxyacetate and brighteners. The maximum plating rate is 0.0005 in./hr. and it is claimed that thicknesses can be controlled to $\pm$0.00001 in.

Advances in electroplating include black chromium-base electroplating,[34] D-chromium plating using tetravalent chromium electrolytes,[35] and a new bronze alloy plating process, *Lustralite*.[36] The solution for electrodepositing the black chromium finish consists mainly of chromium with a small amount of vanadium and, according to M. F. Quaely, meets the demand for a finish possessing good adherence, heat resistance and black body properties. It is claimed that the *Lustralite* copper/tin alloys have given better results than solid bronze on some bearing surfaces and in addition find useful application for decorative purposes, electroforming and for 'stopping off' for surface hardening.

Metallurgical instruments

Instruments recently developed by B.I.S.R.A. include a cheap, simple instrument—the 'Profiloscope'[37]—for examining die angles and die wear, and a photo-electric continuously recording dust meter.[38] The latter, which is manufactured by Radiovisor Parent Ltd., London, enables a continuous quantitative record to be kept of dust content in blast furnace gas and can, of course, be calibrated in conjunction with standard gravimetric methods and to suit site conditions. From America comes news of an instrument developed by North American Philips Co. Inc. and the U.S. Steel Co. for the *x*-ray thickness testing of tin plate.[39] *X*-rays, directed upon the tinned steel, penetrate the tin, and the underlying steel emits fluorescent rays, the intensities of which are measured by Geiger counters. An accuracy of $\pm$2% is claimed. Other new instruments and measuring techniques are the Russian announcement of an ultrasonic microscope,[40] a new French photo-



[*The Distillers Co. Ltd.*

The 'CeDeCut' technique, which employs carbon dioxide as a coolant in machining operations, is based on the formation of solid carbon dioxide ('dry ice') at −109°F. by the sudden expansion of liquid CO_2 close to the cutting edge of the tool, and makes use of the intense cooling power of the solid CO_2 to maintain the strength of the tool tip. The method is stated to be particularly valuable where tungsten-carbide-tipped tools are involved. It is claimed that, using this technique, the life of the tool between regrinds is greatly increased and, at the same time, much higher machining speeds can be used. The photograph shows the 'CeDeCut' technique applied to a +GF+ copying lathe.

electric cell pyrometer,[41] an electronic conductivity meter,[42] first developed in Germany and now available in the U.S.A. by the Magnaflux Corp., and an interferometer method for the evaluation of corrosion.[43] It is claimed that corrosion to a depth of as little as 1 or 2 ten-millionth of an inch can be detected.

NON-FERROUS

Titanium is still very much 'headline news.' It is without a doubt the topic which during the last year or so has again received most attention in the sphere of non-ferrous metallurgy. In fact, so much new information has been released that, in the short space available in this report, reference can only be made to the more important publications.

The total U.S. capacity for the production of titanium sponge continues to increase rapidly and is expected to reach 60 tons/day by the end of 1955.[44] Scrap, however, remains a bit of a problem as, although it is repurchased by the suppliers at $1.50 to $2 per lb., present melting methods are not suitable to remelt it.

Some excellent comprehensive reviews of the metal and its alloys have appeared,[45–47] including one from both the Royal Aircraft Establishment[48] and the U.S. Atomic Energy Commission.[49] From these it appears that the properties of the pure metal which are so attractive are its good corrosion resistance, high strength and ductility, low density, high endurance limits and the high strength of some of its alloys at 450 to 500°C. The solvent power of the molten liquid and the cost and difficulty of fabrication of its alloys, however, remain serious disadvantages. In connection with the latter, the turning, milling, machining and grinding of titanium have been carefully studied,[50] and some details have been given of the General Electric Co.'s practice with alloys Ti-150A and Re-130B.[51] Among the factors which make the metal so difficult to machine are high bearing pressure between chip and tool, poor thermal conductivity of the metal and a tendency for the tool materials to dissolve in the titanium chips.

Other work of essentially practical importance includes that described by N. A. DeCecco and H. M. Meyer[52] on brazing, where silver was found to be a satisfactory filler material when used with a dry powdered flux (AgCl 10%, KCl 36%, LiF 9%, NaCl 45%) and an oxy-acetylene torch; the establishment of conditions suitable for the electrodeposition of 0.005-in. thick copper coatings on titanium;[53] the sodium hydride[54] descaling of hot rolled titanium sheets (even on sheets as thin as 0.015 in.); recommendations for satisfactory spot welding and inert gas tungsten-arc welding procedures;[55] and investigations of the dry seizing and galling characteristics of titanium and its alloys.[56] The dry friction coefficients of titanium and its alloys were found to be unusually high compared with Babbitt metal, aluminium and steel against steel, that for titanium/titanium being higher than that for titanium/steel. Finally, studies have been made of the nitriding of titanium using ammonia[57] and the texture of the cold rolled metal.[58]

On the more academic side, A. E. Jenkins[59] has studied the oxidation of the metal at high temperatures in an atmosphere of pure oxygen and detailed investigations have been made of the following systems: Ti-Ag,[60] Ti-Mn,[61] Ti-Mo,[62] Ti-Cu (also -Ag and -Au),[63] Ti-H,[64] Ti-V,[65] Ti-Cr,[66] Ti-Fe,[67] Ti-Al-Cr,[68] Ti-Al-Mo,[69] Ti-Cr-Fe,[70] Ti-Ta-C,[71] Ti-Cr-Mo,[72] Ti-Al-Sn[73] and Ti-Fe-O.[74]

Molybdenum

Increasing the operating efficiency of gas turbines is very largely a matter of raising the operating temperature, and this has naturally led to a demand for materials capable of withstanding increasingly higher temperatures. Some authorities consider that no considerable increase is possible in the operating temperature of the iron- and nickel-base alloys now in use and, in the race to develop alloys having a useful creep resistance at 1,000°C., metals such as molybdenum, tantalum and tungsten, with melting points above 2,000°C. and which are available in sufficient quantities, are receiving attention. In consequence, such factors as the forgeability, creep strength and ductibility of molybdenum and some of its alloys,[75] casting,[76] pressure welding[77] and even methods of electroplating molybdenum[78] have been investigated. Duckworth[79] has also described successful deep drawing tests on molybdenum sheet, 0.10 in. thick, which was drawn into cylindrical cups with a length: diameter ratio >2: 1, using a special technique. Alloy tool steel or wrought aluminium bronze tools designed to keep the material under tension were employed with Droyt Super TRA lubricants.

Zirconium

Zirconium is probably the metal which has been the subject of most publications by the U.S. Atomic Energy Commission during the period under review. Their method for the production of zirconium by the electrolysis of K_2ZrF_6 in molten sodium chloride has been extended. The most satisfactory operating conditions are stated to be at 820°C., when a current efficiency of 69% and a yield of 80% is claimed, giving a product with oxygen and nitrogen contents of 0.06% and 0.002% respectively.[80] Tests have been carried out to determine the mechanical properties of the unalloyed metal at ambient,[81] moderate and low temperatures.[82] They have performed extensive work on the development of zirconium-base alloys and

have released their 13th, 14th and 15th[83] quarterly reports on this aspect of zirconium metallurgy. Information on the zirconium-aluminium[84] system has appeared elsewhere. Finally, experiments on the anodising of zirconium and other transition metals are reported.[85] Using titanium, zirconium, hafnium, niobium, tungsten and tantalum, voltage/current/time curves were determined in 70% nitric acid at 25°C. with a current density of 1 ma./ohm.[2] Zirconium was found to be the only metal failing to give a well-defined maximum voltage. The black oxide layer formed did not reach a limiting thickness and was not an effective capacitive insulator. Further experiments by R. D. Misch and W. E. Ruther[86] under similar conditions showed that, in nitric acid solution containing >14 wt.-% acid, zirconium showed periodic changes of resistance with time, the transition depending on grain orientation and metal texture. As the acid concentration was increased, the anodic layer changed in colour from white→grey→black. The white oxide was friable and developed from a thin barrier layer; the black oxide was hard and coherent.

According to Schickner, Beach and Faust,[87] zirconium and its alloys cannot be soldered, brazed or hot-dip coated by the conventional methods, as the surface film is not removed by ordinary fluxes. New methods involving a dip in molten zirconium chloride have been devised. Satisfactory a.c. and d.c. welds have been made in zirconium sheet using standard argon-arc equipment or plastic containers flushed with argon to exclude all air from the welding zone.[88]

Miscellaneous

Methods of plating aluminium, copper, nickel, iron, zinc, silver and tin directly on to beryllium have been described by J. G. Beach and C. L. Faust.[89] An alternative procedure involves the formation of an immersion zinc coating on which copper, nickel, iron or zinc are plated directly and chromium over a preliminary copper deposit. Other items of interest appertaining to beryllium include a study of its extrusion[90] (optimum temperature 1,090°C.); the welding or brazing of beryllium to itself and other metals;[91] and the publication of a document with over 100 pages containing 249 references to all aspects of the toxicology of the metal and its compounds over the years 1907-53.[92]

In the sphere of aluminium technology, E. Emrich[93] has described experiences with the use of aluminium foil and powder for the covering and treatment of wounds and ulcers; aluminium 'yarns' are finding some applications in dress or furnishing fabrics;[94] and a new aluminium material, *le Granal*, of interest to architects and decorators is being used in France.[95] The metal yarn is made from aluminium foil in the form of coiled strip, which is slit to the desired narrow widths (1/16 to 1/100 in.)—the latter width being approximately equivalent to that of 20s cotton yarn. It is claimed that the strength of the slit material, even at its finest, is sufficient for it to be twisted with other types of yarn without difficulty. *Le Granal* is 99.99% aluminium that has been given a special rolling sequence and a high temperature anneal. As a result, a material is produced having a mixed macrostructure of large and small grains without directionality. When etched in a mixture of equal parts hydrochloric acid, nitric acid and water with some hydrofluoric acid, the grain structure is revealed and this provides a most attractive finish when anodised.

Another topic of general interest which has hitherto been the subject of much controversy concerns the blackening of aluminium plant and utensils in use under ill-defined circumstances. Although all authorities have agreed that it is harmless, it is a bad advertisement for the metal and the recent investigation by D. Altenpohl[96] is long overdue. He has found that, within ordinary limits, the purity of the metal and the hardness of the water have little effect. The blackening can be overcome by (*a*) boiling in distilled water for 60 to 80 min. or (*b*) heating at 80 to 90°C. in slightly alkaline water (*p*H 11) for 1 hr.

REFERENCES

[1] *J. Iron & Steel Inst.*, 1954, **176** (2), 167-172.
[2] *Ibid.*, 1954, **177** (2), 229-233.
[3] *Ibid.*, 1954, **176** (2), 173-216.
[4] CHEMICAL & PROCESS ENGINEERING, 1953, **34** (12), 384-387.
[5] Amer. Soc. Met., 'Boron Steel,' 2nd revised edition, 1953, pp. 67-69.
[6] *Iron & Coal Trades Rev.*, April 17, 1953, **166**, 871-881, 884.
[7] *Bull. Cercle Etudes Mét.*, Sept. 1953, **6**, 165-200.
[8] *Metal Progress*, March 1953, **63**, 97-104.
[9] *Iron Age.*, Oct. 29, 1953, **172**, 94-97.
[10] Amer. Soc. Mét., 'Boron Steel,' 2nd revised edition, 1953, pp. 61-65.
[11] Amer. Soc. Metals, 35th Annual Convention Preprint No. 23, 1953.
[12] *Rev. Met.*, Feb. 1953, **50**, 73-94.
[13] *Metal Progress*, Aug. 1953, **64**, 78-83.
[14] *Amer. Inst., Min. Met. Eng. Proc. Nat. O. H. Comm. 35th Conf.*, 1952, **35**, 132-133.
[15] Amer. Soc. Metals Preprint No. 21, 1953.
[16] *Ibid.*, No. 20, 1953.
[17] *S.A.E.J.*, Sept. 1953, **61**, 24-27.
[18] *Welding & Metal Fab.*, Nov. 1953, **21**, 425-429.
[19] *Stanki i Instrument*, 1952 (4), 31-32.
[20] *Mech. Eng.*, Sept. 1953, **75**, 701-705.
[21] *Trans. Amer. Soc. Mech. Eng.*, May 1953, **75**, 549-559.
[22] *Tekn. Tidskr.*, Nov. 17, 1953, **83**, 881-886.
[23] *Engineer*, Jan. 29, 1954, **197**, 181-182.
[24] *Machinist*, Jan. 2, 1954, **98**, 1-6.
[25] *Sci. Lubrication*, Nov. 1953, **5**, 18-26; Dec., 18-22.
[26] *Metal Progress*, Dec. 1952, **62**, 71-76.
[27] Inst. Mech. Eng. Preprint, Feb. 20, 1953.
[28] *Rev. Gén. Méc.*, June 1953, **37**, 185-187.
[29] *J. Iron & Steel Inst.*, 1953, **175** (4), 381-390.
[30] *Mat. Methods*, May 1953, **37**, 96-100.
[31] *Metal Ind.*, July 17, 1953, **83**, 47-48.
[32] *Metal Finishing*, Sept. 1953, **51**, 68.
[33] *Sheet Metal Ind.*, Jan. 19-21, 1954, **31**, 30.
[34] *Proc. Amer. Electroplaters Soc.*, 1953, **40**, 48-52.
[35] *Metalloberfläche*, Oct. 1953, **7**, B156-B160.
[36] *Steel*, Dec. 21, 1953, **133**, 102-109.
[37] *Metal Ind.*, May 22, 1953, **82**, 417-418.
[38] *Engineering*, Jan. 29, 1954, **177**, 146.
[39] *Non-Destructive Test*, July 1953, **11**, 33-36.
[40] *Doklady Akademii Nauk S.S.S.R.*, 1949, **64** (3), 333-335.
[41] *Comptes Rend.*, Oct. 28, 1953, **237**, 975-977.
[42] *Mat. Methods*, Oct. 1953, **38**, 124-126.
[43] *Metal Ind.*, Nov. 6, 1953, **83**, 386.
[44] *S.A.E.J.*, 1953, **61** (5), 20-24; (6), 56-65.
[45] *Metal Treatment*, 1953, **20** (93), 245-253; (94), 327-334.
[46] *Ibid.*, 1953, **20** (88), 30-34.
[47] *J. Roy. Aeronaut. Soc.*, 1953, **57** (508), 189-207.
[48] Roy. Aircraft Establishment Publ., 1952 (RAE-TN-MET-155), 31 pp.
[49] U.S. Atomic Energy Commission Publ., 1953 (CRD-A19-27), 35 pp.
[50] *Modern Metals*, 1953, **9** (2), 88; (3), 42, 44, 60, 62.
[51] *Machinist* (Europ. Edn.), 1953, **97** (10), 628-633.
[52] *J. Metals*, 1953, **5** (9), 1197-1198.
[53] *J. Electrochem. Soc.*, 1953, **100** (11), 485-489.
[54] *Iron Age*, 1953, **172** (25), 137-139.
[55] *Weld. J.*, 1952, **31** (11), 1035-1041.
[56] U.S. Atomic Energy Commission Publ., 1953 (NP 4698), 24 pp.
[57] Amer. Soc. Metals Preprint No. 3, 1953, 26 pp.
[58] *J. Metals*, 1953, **5** (10), 1378-1382.
[59] *J. Inst. Met.*, 1954, **82** (5), 213-221.
[60] *Ibid.*, 1954, **82** (5), 222-226.
[61] Amer. Soc. Metals Preprint No. 7, 1953, 16 pp.
[62] *Ibid.* No. 8, 1953, 18 pp.
[63] *Z. Metallkunde*, 1952, **43** (4), 112-118.
[64] U.S. Atomic Energy Commision Publ., 1953 (KAPL-955), 42 pp.
[65] *Ibid.*, 1952 (ISC-228), 165 pp.
[66] *J. Inst. Met.*, 1954, **82** (9), 433-439.
[67] U. S. Atomic Energy Commission Publ., 1952 (AF-TR-6597), 47 pp.
[68] Amer. Soc. Metals Preprint No. 6, 1953, 28 pp.
[69] *Ibid.* No. 5, 1953, 22 pp.
[70] *J. Metals*, 1953, **5** (9), 1209-1213.
[71] *Ibid.*, 1953, **5** (9), 1205-1208.
[72] *Ibid.*, 1953, **5** (11), 1544-1548.
[73] *Ibid.*, 1954, **6** (1), 25-29.
[74] *J. Inst. Met.*, 1954, **82** (11), 534-538.
[75] *Ibid.*, 1954, **82** (8), 345-360.
[76] *Ibid.*, 1954, **82** (8), 361-373.
[77] *Ibid.*, 1954, **82** (8), 374-378.
[78] *Plating*, 1953, **40** (10), 1126-1131.
[79] *Machinery* (Lond.), 1954, **84** (2153), 389-390.
[80] U.S. Atomic Energy Commission Publ., 1952 (NYO-3119), 32 pp.
[81] *Ibid.*, 1952 (SEP-87), 23 pp.
[82] *Ibid.*, 1953 (SO-2510), 42 pp.
[83] *Ibid.*, 1953 (SO-2508), 2 pp.; (SO-2509), 5 pp.; (SO-2511), 7 pp.
[84] Amer. Soc. Metals Preprint No. 13, 1953, 18 pp.
[85] U.S. Atomic Energy Commission Publ., 1952 (AECU-2310), 22 pp.
[86] *J. Electrochem. Soc.*, 1953, **100** (12), 531-537.
[87] *Steel*, 1953, **132** (18), 118-119.
[88] *Murex Rev.*, 1953, **1** (11), 245-261.
[89] *Metal Finishing*, 1953, **51** (10), 68-70, 73.
[90] *Metal Progress*, 1952, **62** (6), 82-84.
[91] U.S. Atomic Energy Commission Publ., 1953 (BMI-836), 38 pp.
[92] *Ibid.*, 1953 (Y-975), 111 pp.
[93] *Aluminium*, 1953, **29** (3), 110-112.
[94] *Textile World*, 1953, **103** (9), 134-136.
[95] *Rev. Aluminium*, 1953 (195), 17-19.
[96] *Aluminium*, 1953, **29** (9), 361-370.

Increased Use of Nickel

Increased demand for nickel for use in the expanding electronics industry is forecast by Dr. John F. Thompson, chairman of the International Nickel Co. of Canada Ltd., in a statement to shareholders. Comparatively small quantities of nickel and nickel alloys are used in individual components of electronic devices, but the production of such devices is so large that the company supplied this industry with more than 7 million lb. of nickel during the past year.

Chemical Engineering in Britain

PROGRESS AND PROSPECTS

'Never was the work of the physical chemist of more significance or indeed more essential than it is now, and never have chemists had greater need of the support of engineers. Never more insistent was the need of the knowledge of scientific principles and of what the Greeks called "nous." Never was it more essential that this knowledge and skill should be present in one brain. . . . There is an insistent demand for chemical engineers whose field is as clear and distinct in this chemical world as that of the civil, mechanical or electrical engineers with whom they will so often have to work.'

These observations were made by Mr. Stanley Robson, M.Sc., D.I.C., F.R.I.C., M.I.M.M., during this year's presidential address to the Institution of Chemical Engineers. As well as surveying the Institution's recent activities, Mr. Robson presented a concise picture of the advance of chemical engineering in this country and of the prospects for the future. Here are some points from his address.

THE biggest chemical engineering developments of our time are in the field of petroleum refining, with its associated petrochemical industry; in processes for the extraction of some of the less common elements; and in the extraction of some of the commoner elements from leaner ores.

The products of the petroleum industry amount already to more than half the weight and value of the chemical industry's, while the weight of metalliferous ores treated and the effort expended in exploration, particularly in the search for materials useful in the field of atomic energy, is very great.

Trend towards larger units

A common characteristic of these new developments is the greatly increased size of the operations. The future is indeed likely to be characterised by much larger units than ever before. An illustration of the trend towards much larger units in the chemical industry can be found in sulphuric acid manufacture. In 1914 a contact acid unit having a capacity of 12 tons/day of sulphuric acid was considered to be large. The Badische-type units erected in this country at that time were of this size, and were the culmination of most costly researches by that company. During the first world war some large double units with two 12-ton converters were built at a number of Ministry of Munitions factories, amongst which was a 10-unit installation at Avonmouth. Between the two world wars, the Avonmouth units were telescoped into six units, each with a capacity of 50 tons/day. During the second world war—elsewhere—new plants were built with unit capacities of 50 to 100 tons/day, and later plants were built with a unit capacity of several hundred tons/day. Today, units of any reasonable dimensions can be built with confidence, but much change of design was necessary before this was possible.

Similarly, roasting furnaces have altered in type and grown in size. Before 1914, in this country, small batteries of hearth roasters designed for 5 tons/day each of pyrites were commonly installed, although many operatives claimed that 3 tons/day was a more convenient size and less troublesome. Such equipment is now far too small, and sizes up to 50 tons/day are preferred. Today, hearth roasting furnaces are themselves largely being replaced by flash roasters which will have a capacity of 100 to 150 tons/day, or by turbulent layer equipment of similar capacity. Even these have been outpaced by sintering machines of which the latest unit has a capacity of over 600 tons/day of zinc blende.

These are merely instances of the trend from small to large units. There are many similar examples in other sections of the general chemical industry. Nothing, however, is more impressive than the growth in size and complexity of petroleum refinery installations. Size and complexity again characterise the processes for the production of fissile materials, and for the recovery of some of the less common elements. In the former the need for effluent restraint and for remote controls have been carried to an extent never before envisaged. The extraction of the less common elements has also called for new methods and techniques. In all of these the chemical engineer has his own special contribution to make. He indeed follows no mean profession.

Table I. Distribution of Members, Associate Members and Graduates of the Institution of Chemical Engineers in the United Kingdom by Industries

*Industries**	%
1. Coke ovens and by-product works (mainly coal tar distillers)	2.8
2. Dyes and dyestuffs	2.6
3. Fertilisers	1.8
4. Synthetic resin and plastic materials	4.9
5. Heavy chemical:	
(*a*) Inorganic	12.2
(*b*) Organic	3.6
(*c*) Industrial gases	0.6
6. Fine chemicals, including pharmaceutical preparations	5.0
7. Explosives	2.9
8. Paint and varnish	0.9
9. Soap, synthetic detergents, glycerine, etc.	2.6
10. Mineral oil refining (mainly petroleum), including petroleum chemicals	11.2
11. Other oils, greases and glues	2.6
12. Chemical plant manufacture	14.4
13. Instruments	0.5
14. Treatment of non-metalliferous mining products other than coal	1.3
15. Metal manufacture:	
(*a*) Ferrous	0.7
(*b*) Non-ferrous	2.4
16. Textiles	3.5
17. Food, drink and tobacco	2.5
18. Paper and paper board	0.6
19. Rubber	0.5
20. Gas	2.6
21. Electricity	0.7
22. Water	0.2
23. Coal	1.8
24. Sewage treatment	0.5
25. D.S.I.R. Research Associations, Stations, etc.	0.9
26. Ministry of Fuel and Power	0.6
27. Private practice	2.8
28. Teaching	3.0
29. Others	6.8

*The *Standard Industrial Classification* has been used as far as possible. Items 1 to 11 are known as the chemical and allied trades.

Wide distribution of chemical engineers

The plant and equipment used in the chemical industry (taking the Association of British Chemical Manufacturers' definition), as measured by the capital employed, has grown by 73% in four years and production has increased by 61% with total employment greater by only 6.7%; also the percentage increase in technically qualified men is 19.9%, whereas the increase in operatives is only 1.7%. These are very significant figures, especially in relation to the Heavy Chemicals Productivity Team report. It is clear that much more productive methods are being used; this major industry is obviously thriving, and confidence is being shown in its development. What about the development of the chemical engineering profession? Is it growing at the same rate? The answer lies in the fact that membership of the Institution of Chemical Engineers has increased from 803 at December 31, 1934, to 3,038 today.

The chemical engineer is employed in many industries other than the chemical industry as defined by the A.B.C.M. Table 1 indicates that slightly more than one half of I.Chem.E. members resident in this country, other than students, are employed in what the *Standard Industria Classification* calls the ' chemical and allied trades,' which include oil refining (items 1-11). The dissection of the remaining industries shows that chemical plant manu-

facturers give employment to 14% of members and that the remainder are widely spread outside the chemical industry. A similar wide distribution of chemical engineers occurs in the United States.

It has been held by some that the responsibilities of chemical engineers in this country are significantly different from those in the United States and that consequently our professional training should be different. In fact, there is a close occupational similarity. There are, of course, some differences, for example in the U.S. only 11% of the chemical engineers are employed in management and administration as against 20% in the United Kingdom, while 31% are employed in research and development in the U.S. as against 22% in the U.K. These figures require more analysis than is possible at present, but it is clear that chemical engineers are well represented in the higher management of the industries which they serve in this country, and that they are making a substantial contribution in the field of research.

Training of chemical engineers

In 1934 the only courses recognised by the Institution were those at University College, Imperial College and King's College in London and at the University of Birmingham. Facilities are now available in more than 20 colleges. There are four Chairs of Chemical Engineering and the University of Durham has taken steps to provide another. It is gratifying also to learn of proposals to establish an endowed Chair of Chemical Engineering in the University of Nottingham and in Manchester, and there is no doubt that others will follow.

In 1950 the Hankey report gave some figures for chemical engineering students in training, and in 1953 the Institution carried out a survey of chemical engineering students in full-time training in universities and technical colleges. A summary of the results of this survey is given in Table 2.

Taking into account the higher degrees, the total number qualified in 1953 was 231, and the total number likely to qualify in 1954 is 293. The corresponding figures in the Hankey report are 195 and 202, but the Hankey report does not appear to take count of higher degrees.

In comparing present capacity with current enrolment, courses should be considered in relation to the organisation on which they are based. For example, in Birmingham University, Cambridge University, Imperial College and University College, London, where there are Chairs of Chemical Engineering, the totals are:

	Annual capacity	*Enrolment 1953-54*
First degree	108	104
Post-graduate diploma ..	55	67

One of these departments has currently 250 applications for the approximate 50 places which are available. There is reason to believe that a substantial number who are not offered places in these chemical engineering departments will read other subjects rather than enrol in places outside the university. This requires serious consideration.

These brief remarks on training will not be complete without mention of the facilities provided by some Government departments and industrial concerns for the practical training of both graduate and undergraduate chemical engineers. Vacation training courses have now been developed to an extent where the majority of chemical engineering undergraduates can obtain an introduction to practice by this method. A number of industrial establishments have well-planned training schemes for the purpose of bridging the gap between the college engineering department and responsible professional work.

Table 2. Survey of Chemical Engineering Training Facilities in Great Britain*

University courses (including first degree courses at Battersea Polytechnic, Bradford Technical College, the Royal Technical College, Glasgow, Manchester College of Technology and West Ham College of Technology, and the post-graduate diploma course at Manchester College of Technology)

	First degree	*Post-graduate diploma*
Capacity	218	101
Enrolment for session 1953-54	186	99
Qualified in 1953 ..	116	65
Likely to qualify in 1954 ..	127	84

Technical college courses

	Degree type	*Post-graduate type*
Capacity	68	65
Enrolment for Session 1953-54	24	10
Qualified in 1953 ..	14	8
Likely to qualify in 1954 ..	19	10

Higher degrees

Enrolment for Session 1953-54 ..	53
Qualified in 1953	28
Likely to qualify in 1954 ..	53

*The figures throughout the report refer to the number of students or candidates.

New British Standards

Lunge nitrometers (B.S. 2070: 1954, 2s. 6d.). Three sizes of Lunge nitrometer, suitable for use in the estimation of nitrogen in nitrates, of total capacities, 50, 100 and 130 ml., are specified. Full details of dimensions, construction, graduation and inscriptions are specified and illustrated. Recommended dimensions for the levelling tubes and stopcocks are given and the standard also includes a leakage test and a method for the determination of capacity.

Recommended names for chemicals used in industry (B.S. 2474: 1954, 10s.). This standard comprises two parts, the first and larger giving recommendations on the names to be used for chemicals and the second part giving a list of recommended names for grades of certain of these. Names which are undesirable on grounds of safety, ambiguity, etc., are listed as 'deprecated' and others, which may in some cases be systematically correct but which are not recommended for industrial use, are given as 'non-preferred.' The list does not purport to be complete, but where chemicals which form the basis for extensive trade are omitted, it is generally because a related compound will provide a guide to the naming of them.

Synthetic resin (phenolic) moulding materials (B.S. 771: 1954, 6s.). This is a revision of B.S. 771, which was originally published in 1938 and the first revision of which was published in 1948. The present revision involves only small changes in levels of quality, generally towards higher quality.

The most important change is the introduction of a simpler statistical technique without loss of discrimination. One value, L, is specified against which is compared the average value of a specified number of tests results on a single batch of material. Another value, X, is specified against which is compared the average value of the results derived from a sequence of batches. The values of L and X are given in the standard. A simple procedure is outlined for determining the specified value appropriate to any number of batches forming a sequence.

Universal decimal classification for rubbers, plastics, stone, etc. (B.S. 1000: 1954, 15s.)—'(Universal Decimal Classification) for Machromolecular Materials (Rubbers and Plastics), Stone and Other Processable Materials.'—This new section of the fourth international edition of the 'Universal Decimal Classification' (UDC) embodies a complete rearrangement of the existing classification schedules for rubbers and plastics, as set out in the third international edition (German) published in 1948. It also includes a short section 679 on the industries associated with stone and other processable materials, such as amber and corals.

Recent advances in the synthetic rubber and plastics industries have rendered obsolete and inadequate the earlier UDC schedules, making it desirable to regroup plastics (from 679.5) with rubbers, natural and synthetic, all under 678. Such drastic transfers are especially inconvenient for general users of the UDC, but this regrouping is regarded as essential for a sound classification of up-to-date information on macromolecular materials by specialist users, for whom primarily this new more flexible notation has been devised.

The chief innovation is for the polycondensates, for which a new 'sign of synthesis' (*apostrophe* or *quote*) has had to be introduced to specify individual compounds containing the 'essential groups,' these being regarded as the components interacting to form specific polycondensates.

SILICONES

Full-scale Production in Britain

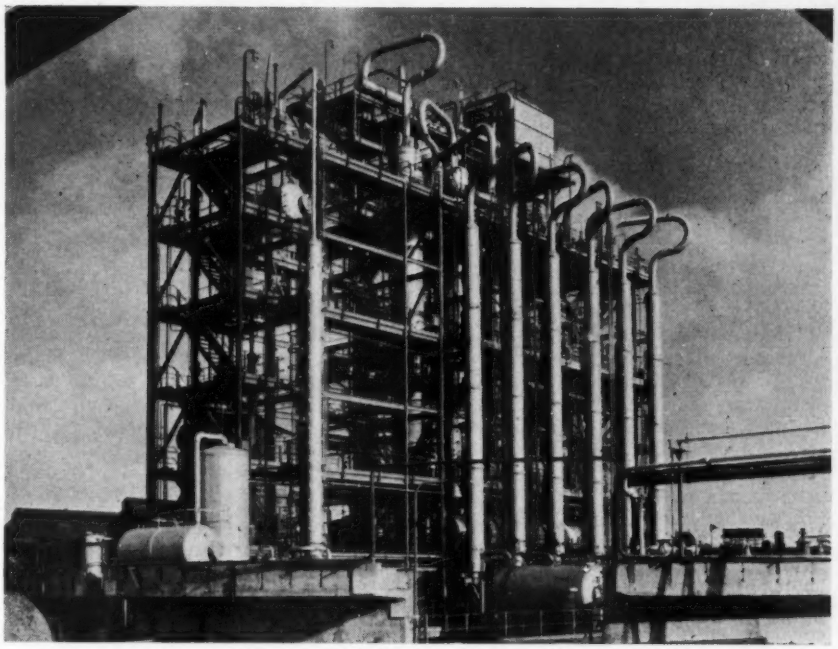
Commercial production of silicones has begun at the new £1-million factory of Albright & Wilson Ltd. at Barry, Glamorgan. The first full-scale silicone plant in the U.K., it has a capacity for producing 600 tons p.a. of silicones and Britain will no longer have to depend on America for its supplies of these versatile materials. Since the manufacture of silicones involves the handling of hazardous and highly reactive raw materials, the plant has several unusual features. Some of these features are noted briefly here, along with an account of the processes used and the background to this new enterprise.

SILICONES are organic compounds of silicon, available in the form of fluids, greases, resins, rubbers, etc., which have many useful properties including stability to extremes of temperature and to oxidation, good electric properties, water-repellency, low surface tension, etc. Albright & Wilson Ltd. recognised the importance of these new materials when they were introduced to the U.K. in 1945; by 1946 they had arranged with Dow Corning Corporation, of the United States, to import their silicones into this country.

Even at that time it was implicit that Albright & Wilson would make silicones here and, in 1950, a suitable site was found and purchased at once. It was the plant and site of the Ocean Salts Manufacturing Co. Ltd. at Barry, Glamorgan. This plant had been erected during the war for the extraction of magnesium salts from sea water and ceased production shortly after. The office and laboratory blocks could be put into use immediately and certain other buildings were found readily adaptable. Use was made of the great concrete settling tanks for bund walls to the tank farm and distillation plant. Extensive repairs have been made to the buildings. The silicone plant itself is, of course, entirely new. The site covers 26 acres.

The plant was designed by the company's own central engineering department and based on full working drawings supplied by Dow Corning.

The factory has been built by the central engineering department of Albright & Wilson Ltd., but certain special work has been contracted out.



The distillation unit, shown here, is a vital part of the new plant for producing silicones at Barry, South Wales. In this view, the still pots and batch columns can be seen.

Basic reactions

There are two main industrial methods of synthesising chlorosilanes, the intermediates necessary in the production of silicones. Both methods are used at Barry.

The first method is known as the Grignard process. Silicon tetrachloride, prepared by reacting silicon with chlorine gas at about 1,000°C., undergoes a series of reactions with organic chlorides in the presence of magnesium metal in ether to produce a mixture of chlorosilanes, *e.g.*:

$$SiCl_4 + RCl + Mg \longrightarrow RSiCl_3 + MgCl_2$$

In the second process, the 'Direct method,' the vapours of organic chlorides are passed over silicon metal at about 300°C. in the presence of small amounts of copper or silver as catalysts. The organic halides react with the silicon to produce chlorosilanes, *e.g.*:

$$Si + 2RCl \longrightarrow R_2SiCl_2$$

In both processes, mixtures of mono-, di- and tri-substituted chlorosilanes (*e.g.* $RSiCl_3$, R_2SiCl_2 and R_3SiCl) are formed, and these must be separated by fractional distillation. The separation is difficult because the boiling points of the chlorosilanes lie very close together. Thus fractionating equipment of great efficiency has to be used.

The next step is the hydrolysis of these chlorosilane intermediates by reacting them with water. The products of the hydrolysis condense and by varying the initial proportions of the different chlorosilanes a great variety of products can be made. These are silicones.

$$R_2SiCl_2 + 2H_2O \longrightarrow R_2Si(OH)_2 + 2HCl$$

condensation

$$n R_2Si(OH)_2 \longrightarrow \left[\begin{array}{c} R \\ | \\ -Si-O- \\ | \\ R \end{array}\right]_n + nH_2O$$

a linear silicone polymer

Materials and processing problems

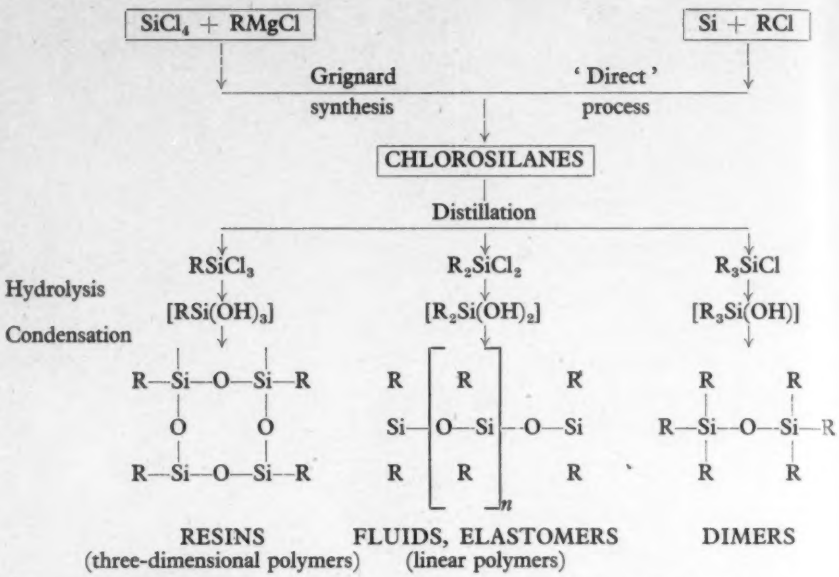
The most important raw materials used are silicon metal and methyl chloride. In addition, considerable quantities of ether,

magnesium, copper and solvents are required. The silicon metal has to be as pure as possible, in contrast to the 30 to 80% purity sufficient for its use in alloy steel.

Some chemicals involved in carrying out the reactions are highly inflammable. The chlorosilanes are corrosive, will react spontaneously with water, are toxic and are also highly inflammable.

The processes employed are complex and need to be carried through many stages with great care in order to produce pure materials in good yield. The Grignard process, for instance, has hitherto been considered a laboratory method of synthesis and its successful employment on a large scale is a considerable feat of chemical engineering.

The handling of the hazardous and highly reactive intermediate products and solvents used in silicone production presents considerable difficulties. In all stages in the production of the chlorosilanes, which includes the distillation process, the entire system has to operate in anhydrous conditions and thus the plant has had to be designed to ensure the complete exclusion of atmospheric moisture.



Reactions involved in the commercial preparation of silicones.

Features of plant

To produce commercially useful products, the various chlorosilanes must be obtained very pure. This requirement has called for the design of fractionating columns of great height and many stages. The distillation plant is controlled by automatic instruments indicating a graphic panel in the control room which is said to be one ot the largest of its kind in Europe. The system of control valves and indicators has been so arranged that signals are given simultaneously on the plant and on the control panel.

The plant successively takes off low-boiling and high-boiling materials and then the monomethyl and dimethyl bodies. Six columns are provided to handle by-products and there are three small columns to purify imported ether for the Grignard process.

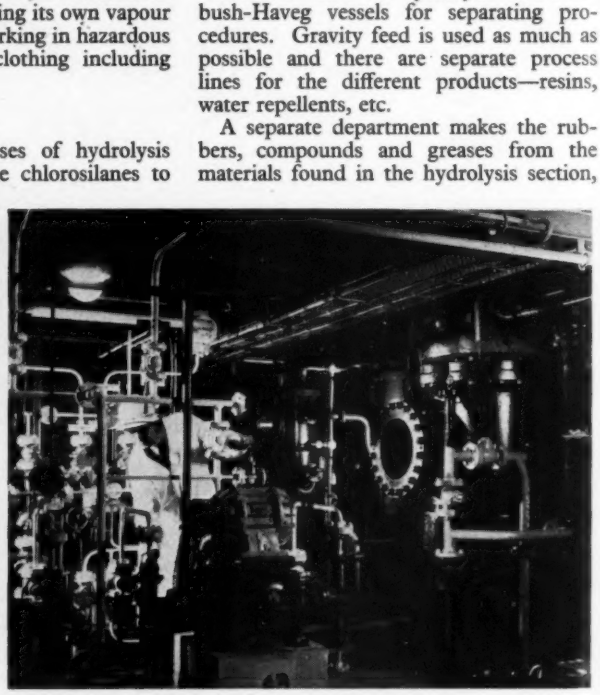
Elaborate safety precautions are necessary and the volatile solvents and intermediate products of the process are stored in buried tanks. The tanks, and indeed all parts of the system, are purged with inert gas to prevent the ingress of air or moisture and to reduce the risk of fire. Incoming solvents are transferred to the storage tanks from the tank cars in which they are transported by pumps. On the other hand, methyl chloride, which is also transported by road tanker, is transferred to storage tanks by utilising its own vapour pressure. All people working in hazardous areas wear protective clothing including glasses and helmets.

Hydrolysis unit

The essential processes of hydrolysis and condensation of the chlorosilanes to form silicones take place in the hydrolysis unit. Points of particular interest here are the extent to which all the equipment has had to be made flameproof, the way in which the high building has been made use of and the room that has been left for expansion.

The hydroxyl bodies produced in the hydrolysis plant are the siloxanes or silicones. They are washed and polymerised and then blended to produce fluids of different viscosities. Finally, they are polished in filter presses. Glass-lined vessels are used for hydrolysis and Keebush-Haveg vessels for separating procedures. Gravity feed is used as much as possible and there are separate process lines for the different products—resins, water repellents, etc.

A separate department makes the rubbers, compounds and greases from the materials found in the hydrolysis section,



Some further views of the Barry plant. *Left:* **The tank farms.** *Right:* **Part of the chlorosilane production unit.**

and here considerable precautions are taken to avoid contamination with organic materials. All the operations are carried out with the most rigid controls to keep the products within the narrow limits of their specifications.

In view of the recent announcements about the production of synthetic rubbers in this country, it is interesting to note that silicone rubber was the first synthetic rubber to be made in the U.K.

Water services

Water supplies for the works are drawn from two sources. Cooling water is pumped from the Cadoxton River, which is one of the boundaries of the site, and recirculated through a cooling tower. It has to be filtered, chlorinated and softened. Town's water is taken from the local supply with its reservoir in the Brecon Hills. This water is extremely soft and needs only a little Calgon treatment before being suitable for steam raising.

Market prospects

The distribution of Albright & Wilson's silicone products is in the hands of Midland Silicones Ltd., which is a subsidiary company owned jointly with Dow Corning Corporation. Midland Silicones Ltd. was formed in 1950, Albright & Wilson having undertaken distribution themselves since 1946.

Since their first introduction into this country, silicones have become familiar to many industries, and are used as silicone rubber gaskets, greasy packings, valve and cock lubricants, antifoaming agents, silicone paints, release agents, etc., to name a few of the industrial uses.

The export market for silicones has expanded rapidly over the past two years, primarily in the Commonwealth and European countries. With increasing supplies of British-made silicones, further markets are at once thrown open and therefore an even faster expansion is to be expected.

Calcium Hydride's Promising Future

By A. G. Arend

THE extent to which calcium hydride may be used industrially in the near future shows still greater possibilities than was at first anticipated. This applies alike to uses in organic chemistry, inorganic chemistry, and metallurgy. In respect of reactions of the kind used to prepare isophorone from acetone or crotonaldehyde from acetaldehyde, calcium hydride holds advantages over other alternative condensation agents.

It may be used wherever aqueous or alcoholic solutions are to be avoided, and is a mild reagent which does not generally cause repetitive condensations with attendant resin formation. In the prefumery, cosmetic, and associated industries, calcium hydride indirectly is likely to be of service in the preparation of esters and other individual constituents of essential oils, fruit essences, and flavourings. With condensation of esters, this reagent again assists in the acetoacetic ester type of reaction. Since no alcohol need be present, the use of calcium hydride is again considered advantageous, and the reactions can be carried out although solvents are absent, or with high-boiling ethers or hydrocarbons. These reactions have been the subject of intensive researches, although not yet fully developed. In using bivalent calcium derivatives in reactions with dihalides, the advantages possible have by no means yet been exhausted.

With regard to other organic reactions of interest, nitrobenzene is reduced to nitrosobenzene, or even further to azoxybenzene, both of which are useful intermediates in dye chemistry.

By allowing calcium hydride to react with alcohols, a number of promising reagents can be formed. For example, monocalcium ethylene glycolate is contended to be an excellent condensing agent for the type of reaction described above.

Inorganic chemical processes

In inorganic chemical processes, a number of unexpected developments have already arisen, which have opened up possibilities of better reductions and means of separation, etc. For example, zinc fluoride and potassium fluoride may be reduced to their respective metals, by heating with calcium hydride to 500°C.

While the reaction with zinc would at present appear to be more one of academic interest, that with potassium reveals a means of acquiring the metal cheaper than by existing methods. A glance at the current prices of potassium fluoride and metallic potassium will readily show the difference which exists, but production economics depend entirely on acquiring the calcium hydride cheaply.

The fact that sulphates can be reduced to sulphides has already been taken advantage of in the examination of oils for determining the sulphur and sulphates present. The same reduction may be applied to sulphuric acid itself, to convert it to sulphur and hydrogen sulphide. Nitric oxide can be converted to ammonia but the costs again suggest at present that this process is of only academic interest. A different state of affairs seems possible with boron, which like potassium is costly compared with the respective salts. Existing methods of preparing boron involve somewhat elaborate plant, whether produced by the electrothermal, the gaseous, or the costly potassium reduction system. Calcium hydride, on the other hand, reduces boric oxide and borax directly.

Silica can likewise be directly reduced to silicon, in place of the existing electrothermal method. The question of applying these methods profitably appears to be of more account than any doubts as to the capabilities of reduction, since under the proper conditions there are comparatively few materials which cannot be reduced by calcium hydride.

Metallurgical applications

From the metallurgical aspect the possibilities appear to be still greater. Many metals and alloys can be directly prepared from their oxides, sulphides, fluorides and chlorides and this is likely to benefit many of the rarer metal activities. Metallic zirconium, titanium and alloys of titanium with 25% copper, 90% copper, and 20% nickel, made by calcium hydride reduction, have already been marketed. One of the main features is the ease with which oxides, normally difficult to reduce, are dealt with. By using a mixture of metal oxides, the preparation of alloys, which could not be considered possible by powder metallurgy methods is efficiently carried out. In attempting to prepare chromium alloys from mechanical mixture of metal powders, by the usual sintering operation, the particles of chromium oxidise and the mixture is rendered more difficult to alloy. On the other hand, the 100 to 300 mesh powders produced by reduction with calcium hydride are proper alloys, thereby greatly simplifying the sintering process. The pure metals or their hydrides are prepared by heating the metal oxide in an atmosphere of hydrogen with calcium hydride. This gives the metal or its hydride and lime, and where the former is wanted, the mass is cooled in a vacuum, whereas where the hydride is wanted, the hydrogen atmosphere is maintained to the conclusion. The reason why metals produced in this way are particularly suited for powder metallurgy is that every particle has fused, providing discrete spheres which have better physical properties than powders prepared by other means, and which are more stable. Among recent developments are experimental zirconium-lead alloys which are proposed for use as 'flints' for cigarette lighters, and various metal powders for the production of permanent magnet alloys, besides zirconium hydride for use as 'getters' (residual gas adsorbents) in electronic tubes. Other products are different titanium-copper alloys for addition to various non-ferrous alloys for the purpose of reducing porosity, and zirconium-magnesium alloys for refining the grain and reducing the corrosion of magnesium.

Plant and Equipment

Distillation plant

A new distillation plant, which departs from orthodox methods of distillation, is claimed to offer the following advantages:

(1) Uninterrupted production for 365 days a year.

(2) Distillation of any number of different crudes such as tar, crude tar acids, crude bases, naphthalene benzole, benzolised wash oil, alcohols or any other distillable crude in one plant at the same time, and the production of any number of distillates, either commercial or pure.

(3) Operation as a continuous plant, batch plant, or a combination of both at the same time.

(4) Operation under vacuum, atmospheric or elevated pressures at the same time.

(5) Concentration of all the distillation processes on a works into one plant, which only requires one trained operating and technical control staff.

(6) The capacity can be increased without having to install a completely new plant and so cause the existing one to become obsolete.

(7) The heating coil is of steel, and will not coke, corrode or erode. No special steels are required and no joints are in the heating zone.

The distillation of tar presents many difficulties as the crude containing water, solids and free carbon is very prone to decomposition, in addition to having corrosive properties. In the present method of continuous distillation where all or part of the crude passes through the pipe still, the conditions naturally give rise to polymerisation and coking. In the new plant, it is claimed, none of the crude enters the heating unit, and the problems of coking, polymerisation or breakdown of the crude do not arise.

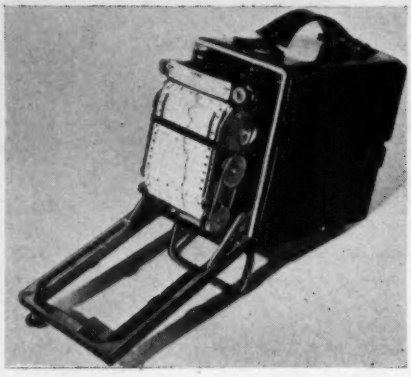
The heat-transfer medium is a specially treated oil, produced within the plant. This oil sets up non-corrosive conditions and enables a mild-steel heating system to be employed and therefore simplifies the normal construction.

The plant consists of any number of differential units according to the number of crudes and fractions to be handled. Each differential unit consists of an independent distillation and fractionating system, which can be brought in and out of circuit at will, and can be used as a continuous unit or batch unit. They can be used in series, or parallel, and arranged to suit any particular requirement, while the plant is in operation.

The plant has been developed by the newly formed Horace Priest Chemical Engineering Co. Ltd. An interesting development is the fractionating column, which has a number of bubble-cap trays, whilst its construction has the simplicity of a packed column; the column is made in one piece and does not contain the usual individual sections, so that joints and flanges are obviated. A further claim is that, owing to the large number of plates in the fractionating equipment, and unlimited reflux ratio, pure products can be obtained. Where fractions can only be obtained by azeotropic distillation, the introduction of a third component into the system can be made without affecting the other crudes being handled.

Unlike orthodox plants, where a large quantity of water is used for condensing the reflux vapours, the heat-transfer medium is used to absorb this heat. In addition, the plant is capable of producing its own process steam.

It is stated that a ground space of only about 70 ft. by 16 ft. is required to produce six fractions and a residue. No buildings or structural work are required and the foundations, apart from the heating furnace, can be simple rafts.



Miniature recorder with front open.

Torque limiters

Protection of machine components against the effects of accidental mechanical overloads is a constantly recurring problem to every machine designer. Fords (Finsbury) Ltd. claim they have a solution to this problem in their new range of torque limiters which is, as far as the manufacturers are aware, the only range of its type being offered in this country ' off the shelf ' to machinery designers.

Each of the torque limiters in the range is said to be easily adjustable to transmit any desired torque within fairly wide limits, and it automatically disengages as soon as the set torque is exceeded through a mechanical overload occurring in the machine drive. This ensures that no part of the machine is damaged through accidental overloads, and consequently no strengthening-up of parts is necessary to withstand overloads, nor are shear pins necessary in any part of the machine, it is claimed. The torque limiter can be used as a coupling in a shaft, or combined with a V-pulley, a chain sprocket or gear drive. It can also be combined with an electric cut-out switch to stop the machine when an overload occurs and the torque limiter disengages the drive.

A feature of the limiters is that, when the cause of the overload is cleared and the machine is restarted, the limiter automatically resets itself in the same angular position, thus allowing the machine to resume operation at the exact cycle point of release. This is, of course, important on some types of machinery—packaging machinery, for instance, where an exact cycle of operations must be maintained.

Miniature recorder

The modern trend in industrial control equipment design has been towards miniaturisation, and Evershed & Vignoles Ltd. have introduced their miniature recorder primarily for use with this type of work. At present this instrument is supplied for the measurement of voltage or current a.c. or d.c., and for temperature measurement operating directly from base-metal thermocouples.

The recorder is of the tapping type, the record being made by an ink-impregnated ribbon brought into contact with the chart by the movement pointer, which is depressed by the chopper bar operating every 5 sec. There are therefore 12 taps and consequently 12 recorded points made on the chart every minute (this is at a chart speed of ½ in./hr.). The chart has straight time lines and hence rectangular coordinates. This feature enables the record to be read easily and to be readily integrated if desired. It also provides for easy comparison between two or more records.

The self-starting synchronous motors can be supplied for 230 v. 50 cycles, 110 v. 50 cycles, or 110 v. 60 cycles operation.

The recorder movements are of the moving-coil and rectifier-operated moving-coil types, and have a sensitivity down to 1 ma. for full-scale deflection with a resistance of 250 ohms. The speed of response is 0.8 sec. for full-scale deflection. The movement is adequately damped.

The maximum self-contained ranges are 500 v. and 10 amp. The maximum set-up permissible is 4 to 5, *e.g.* the chart of a voltmeter may be scaled from 200 to 250 v.

The front of the instrument case measures only 4¼ in. wide by 6¼ in. high. The case depth is 9 in. This case, which is constructed of aluminium, is adaptable for either flush or projecting mounting by the use of special steel straps and, when required as a portable instrument, it is fitted into a leather sling with a carrying handle. The sling, which fits neatly round the instrument, has a folding foot in front so that the recorder may rest in a sloping position for convenience in reading the scale when the instrument is standing on a table.

Chemical and Engineering Research in Australia

Once again, in their fifth annual report, which covers the year ending June 30, 1953, *the Australian Commonwealth Scientific and Industrial Organisation describe a wealth of fundamental and applied studies carried out by their various research divisions and sections. Since, in February* 1954, *we published extracts from the fourth annual report, substantial progress has been made on many of the research projects described. Here, as previously, we summarise those sections of the report which are of especial interest to chemical engineers and industrial chemists. Extracts from the Organisation's first, second and third annual reports appeared in our September* 1950, *November* 1951 *and December* 1952 *issues.*

CHEMICAL ENGINEERING

FUNDAMENTAL studies of particular application in chemical engineering are the investigation of liquid-vapour equilibria, the physical and chemical effects of high pressures and the development of new chemical engineering techniques. These are described hereafter under the heading of physical chemistry. The major investigations in the applied field have been directed to fuel research, but work has also been done on the fluid-bed roasting of alunite, and a start has been made on the construction of a large pilot plant for the conversion of copper concentrates to sulphates by roasting in a fluid bed.

Utilisation of low-rank coal

Investigation of the gasification of Victorian brown coals has continued. Modifications have been made to the coal feed equipment and to the control system of the fluidised-bed synthesis gas pilot plant, and a number of experimental runs have been completed. The analysis of the experimental data from these runs is complicated by the difficulty of determining accurately the water vapour content of the product gas. Many of the proposed methods for carrying out this determination have been investigated experimentally, but a completely satisfactory method has not yet been devised.

The erection of equipment required for the investigation of the production of gas of high calorific value by the pressure gasification of brown coal is nearing completion. The equipment is designed to investigate under continuous steady-stage conditions the reaction between brown coal and carbon monoxide-hydrogen mixtures at pressures up to 50 atm. The aim of this work is to obtain information on the mechanism and rate of methane formation during pressure gasification, and to make a preliminary evaluation of a new two-stage pressure gasification process. The first part of the pilot plant, which has been designed to produce and store up to 3,000 cu. ft. of a hydrogen-carbon monoxide mixture in a given ratio, has been completed and tested. The erection of the second part, the actual reaction system, is well advanced.

The major pilot-plant projects described above are being supported by fundamental studies related to the gasification of coal. The study of the catalysis of the gasification reaction by ash constituents has been continued, and subsequently completed as far as the water-gas shift reaction is concerned. Some work has been carried out on the influence of inorganic constituents on the reactivity of carbon towards steam, and attention is now being given to the catalysis of methane synthesis by ash constituents. Apparatus has been built to study the flow and diffusion of gas within the pores of the powdered solid catalyst and to determine the pore size and distribution in coal-char particles. Some progress has been made on the investigation of the influence of pressure on the kinetics of the carbon-steam reaction, but it has been limited by shortage of staff.

Roasting of alunite. During the year a series of batch roasts was carried out in an 8-in.-diam. fluid-bed roaster to determine the optimum conditions for roasting alunite. The results were encouraging and plans are now being made to convert this unit to enable continuous roasting tests to be performed. It appears likely that yields of potassium sulphate of up to 90% may be obtained by this means; this would make the process more attractive economically. However, considerations of heat economy may make the design of the full-scale roaster somewhat complicated and expensive.

Roasting of copper concentrates. During the year the Organisation entered into a co-operative arrangement with Mount Morgan Ltd. and the Mount Lyell Mining & Railway Co. Ltd. to build and operate a pilot plant incorporating a 3-ft.-6-in.-diam. fluidised bed to investigate the sulphate roasting of their copper concentrates. Both companies believe that very great advantages may result from a major change in their processes for the recovery of copper and precious metals from mill concentrates. The new process involves roasting to obtain most of the copper in a soluble form as copper sulphate, leaching of the calcines and electrolysis of the pregnant leach liquors. The gold and silver remain in the leach residues and may be recovered by cyanidation.

Until the advent of the fluidised-solids technique as applied to roasting, it was not possible to control the roasting conditions sufficiently to obtain the copper consistently in the desired form. Preliminary tests in fluid-bed roasters have given very favourable results, but, because of the large capital expenditure which would be involved in such a major change in their operations, the two companies desire to investigate the process on a considerably larger scale before making a decision. The pilot plant envisaged will roast about 3 tons/day of concentrates.

INDUSTRIAL CHEMISTRY

Minerals utilisation

The current programme is based on some of the less common metals, minerals, and their derivatives and arises partly from the relative abundance of certain of these minerals in Australia and the increasing industrial attention being devoted to them. It is also, in part, dictated by the coexistence of a variety of chemically similar metals, or elements, in several of the minerals under consideration. The separation of these closely associated elements is usually a necessary prelude to further studies on their individual utilisation and the devising of improved methods of separation becomes a major part of the investigation where, as frequently happens, the known methods of separation are imperfect. The various research projects in the current programme, which are briefly reviewed hereunder, provide several examples of this sequence.

Lanthanon and thorium chemistry. The chemistry of the lanthanons, or rare earths, and of the frequently associated element thorium, has occupied attention for a considerable time past. The practical difficulties associated with the separation of most of the lanthanons from the complex mixtures in which they occur have, until recently, strictly limited any consideration of devising industrial uses for certain of them. The recent development of improved separation techniques in overseas

laboratories has been chiefly related to studies on the fission products of uranium and this Section's current work has been directed towards the development and adaptation of such of these modern methods as may be applicable to the problems of industrial separation of the lanthanons and related elements from mineral sources.

The natural occurrences of this large group of closely associated chemical elements are very diverse in Australia, and for the most part the recovery of the minerals, or their chemical derivatives, is subsidiary to the concentration or chemical processing of other minerals. Thus monazite, the main source of the cerium group of lanthanons and thorium, is recovered in the mechanical concentration of titanium and zirconium minerals from the beach sands of eastern Australia and occurs also in many alluvial tin drifts. Minerals of the fergusonite type, in which the yttrium group of lanthanons predominates, are known to occur with tin ore in Western Australia and elsewhere. Certain Australian uranium ores also contain the lanthanon suite of elements as subsidiary components. These three sources have been availed of in the current work on separation techniques which have been mostly based on the formation of complexes with various organic reagents followed by ion exchange.

One aspect of this work has been the subject to a patent application as a new method of lanthanon separation, and several publications dealing with other phases of this work have been prepared. Preliminary investigations into new sources of lanthanons and related elements have involved examinations of a variety of Australian tungsten ores and associated gangue minerals, with the result that epidote from the scheelite deposit at King Island has been shown to contain lanthanons, although the scheelite itself does not. The occurrence of appreciable amounts of the rare element scandium in davidite concentrates from South Australia has been confirmed and small amounts of scandium compounds have been isolated from this source for the first time. This work has indicated that the published chemistry of scandium in complex mixtures needs considerable revision and to this end the investigation will be continued. Fundamental work on the carbides and iodides of thorium relating to the thermal deposition of the pure metal was continued. Work is also proceeding on the fluorides of cerium and their possible use as fluorinating agents.

Titanium and zirconium chemistry. Investigations on the preparation and properties of titanium nitride have been continued. This material, which is derived from rutile *via* titanium tetrachloride, shows promise for the preparation of various industrial hard 'metal' compacts analogous to those composed of tungsten carbide. The many variables involved in the composition, melting, bonding and sintering of the nitride have necessitated a very extensive series of test pieces being made and compared for performance. The possibilities of using titanium nitride as a component of special types of electrodes and as an ingredient of the composite ceramic-metal compositions known as cermets have also been examined.

A series of investigations on the properties and possible uses of other binary compounds of titanium was commenced. The electrical properties of sintered polycrystalline compacts of partially reduced titanium oxide were studied in conjunction with those of sintered titanium oxide rendered photosensitive by introduced impurities.

Work on the chemical separation of zirconium from associated hafnium was continued with satisfactory results. A new process devised for this separation has proved effective in laboratory-scale tests and the equipment necessary for pound-scale tests was designed and assembled.

Uranium ores. Work on the extraction of uranium from the davidite ore from Radium Hill, South Australia, was concluded and four of the Section's staff joined the research staff of the South Australian Department of Mines to continue investigations on the treatment of this ore and related problems. Current work on davidite ore in this laboratory has been restricted to devising methods for the isolation of scandium and recovery of lanthanons from by-products obtained in the treatment of the ore.

Investigations were continued on the treatment of uranium ore from Rum Jungle, in the Northern Territory, with satisfactory results. This work has been done in collaboration with Consolidated Zinc Pty. Ltd. who have supplied ore samples from the mine, and also with the South Australian Department of Mines and the Melbourne University ore-dressing laboratory.

Cement and ceramics

Work has continued on the study of portland cement and pozzolanic cements, on ceramics including refractories, on the survey of Australian resources of ceramic clays and on the structure and properties of the clay minerals.

Cement and concrete. The research programme covers a number of factors that influence the setting and hardening of portland cement and the durability of concrete. Various reactions that take place during the setting of portland cement are being studied, and they are being correlated with actual setting and strength development. This includes the action of accelerators and retarders, and the factors that give rise to 'false' setting and 'flash' setting. In addition, work on the hydration of cement minerals and the development of strength was recently commenced. All these investigations are directed to obtaining information which could lead to the development of stronger, more rapidly acting cements and more durable concrete.

A study of the behaviour of certain finely ground siliceous materials known as pozzolanas, and the development of strength of blended portland-pozzolana cements in mortar has been continued. These pozzolanic materials have considerable value in improving the durability of concrete under certain conditions, and for use in mass concrete such as in gravity dams.

Although much of the mineralogical work on cement clinker has been suspended, the study of the separation of 'glass' from portland cement clinker at high temperatures and determination of its composition has continued. Further studies are also being made of the action of this glass on clinkering and the formation of compounds in the clinker.

Cement-kiln refractories. The work in this field, which is concerned with the investigation of factors which affect the life of kiln linings, includes a study of the attack by potash volatilised from the kiln feed on high alumina and chrome-magnesite bricks. It has been shown that the interstitial bonding material is attacked and that its physical properties are altered. As a result it has been possible to indicate how this form of attack on certain aluminous bricks may be minimised.

Because of the extensive overseas use of bauxite as a raw material for high-alumina refractories, the possibility of making suitable bricks from Gippsland bauxite is being investigated. Though the amounts of certain impurities are higher than is considered desirable, bricks have been made from it and their properties are being evaluated.

The influence of purely mechanical stresses on the life of refractory bricks is also being investigated. A study of the changes of cross-section taking place in the kiln shell during rotation has indicated the nature of the disruptive forces and how they may be minimixed by placing stiffening rings around the shell.

Gas-works refractories. The investigation of factors limiting the durability of gas-works refractories under conditions of heavy steaming has been continued. Under these conditions the life of the retort is reduced by an increased tendency to slagging and cracking. An attempt is being made to diagnose the cause by reproducing these deleterious effects in the laboratory under conditions which simulate those in a retort during operation.

Special ceramics. Attention is being given to special oxide refractories and to the ceramic-metal combinations known as cermets, which have many special uses, such as for high-temperature jets and turbine blades.

Sand for shell moulding. The recently developed foundry technique of shell moulding requires a sand with unusual physical properties. Surveys have been made of two deposits of sand considered suitable for production of shell moulds, and samples have been subjected to standard tests and employed in experi-

mental moulding operations. The advisory service provided by the Section in general foundry problems has involved several minor laboratory investigations, including small-scale casting operations. Work has been continued on production of synthetic moulding sands using Australian bentonitic clays.

Physical chemistry

This research programme comprises physico-chemical studies, both fundamental and applied, of the properties of surfaces, the properties of fluids and the transfer of materials from one phase to another. Fundamental studies include liquid-vapour equilibria of value in distillation problems; the properties and reactions of fluids under high pressure, providing data of value in predicting the properties of liquid and gaseous systems; and the mass transfer of materials across liquid interfaces. On the applied side, set against a background of fundamental surface chemistry, are the selective flotation of minerals, the recovery of wool wax, dropwise condensation in condensers, the control of evaporation by surface films, continuous methods of separation and the principles governing the effectiveness of fillers in rubber.

Properties of liquids. The wide uses of industrial chemical processes involving liquids, such as distillation and solvent extraction, make an understanding of the thermodynamic properties of liquids and solutions of fundamental importance to the chemical engineer. The combined experimental and theoretical study of this problem has therefore been continued. The liquid-vapour equilibria and heats of mixing of the system ethyl alcohol + carbon tetrachloride have been measured and the behaviour of the mixture has been explained theoretically.

A convenient and accurate method has also been developed for the calculation of the composition of the vapour in equilibrium with a solution from the total vapour pressure. A theory previously developed for predicting the properties of solutions containing alcohols has been extended to cover solutions of a number of other 'associated' liquids, many of which are of industrial importance. A further theoretical advance has explained some of the properties of two-phase liquid systems, particularly those which become completely miscible at both high and low temperatures.

Chemical and physical effects of high pressure. The industrial importance of the accelerating influence of high pressures on some chemical reactions is now well established. But the amount of quantitative work which has been done on even simple chemical reactions is surprisingly small. A systematic study has therefore been initiated at the Division's high-pressure laboratory in Sydney of both theoretical and experimental aspects of the chemical effects of pressure. This has established the underlying cause of many of the reported effects of pressure. Measurements are continuing on the displacement of equilibria and the changes in reaction rates brought about by pressures up to 12,000 atm.

Further measurements have been made of the pressure-volume-temperature relationships of gases at both low and high pressures. The volumetric behaviour of hydrogen and deuterium at low temperatures has been shown, both by theory and experiment, to be subject to large quantum effects not found in heavier gases.

The second and third virial coefficients (indicative of the departure of actual gases from the behaviour of an ideal gas) of a number of hydrocarbon gases have been computed from their published compressibilities and a molecular theory of these coefficients for polyatomic molecules in general is being worked out. This theory should be useful in predicting many of the properties under high compression of gases with complex molecules.

Continuous adsorption processes. The work on the purification of antibiotics by two fractional adsorption processes has been concluded. These processes are being followed up by a local manufacturer. Some of the techniques devised for this work have been used in a continuous ion-exchange process with the ultimate aim of applying them in large-scale metallurgical processes. Special importance has been attached to the development of a continuous process. Previous attempts elsewhere have failed because of the use of vertical towers; these cause difficulties in moving a bed of ion-exchange resin in a uniform manner countercurrently to a flow of liquid. Difficulties are also encountered in removing resin uniformly over the whole cross-sectional area, and in avoiding abrasion of the resin. These disadvantages have been overcome by adapting a mineral jig, and the process developed provides ease of control and economy of resin, and it may be used for either stripping or fractionation. A bed of ion-exchange resin retained on a screen in a long, narrow trough is maintained in a non-turbulent semi-fluid condition by a simple pulsating upflow which causes it to move linearly along the trough. Initially water softening was chosen as a process for study because of its experimental advantages. Calcium ions are continuously removed from hard water in a softening unit and the calcium resin is regenerated (after removing excess water on a filter) by a countercurrent flow of brine in a separate unit. The resin is then rinsed, de-watered, and returned to the softener. This process, which is being operated on a pilot scale, appears likely to be adaptable to other operations, and a patent application has been lodged.

Mass transfer. In connection with some work which is being done on the separation of the components of wool wax by solvent extraction, a study is being made of the effect of hydrodynamics in mass transfer of materials from one liquid phase to another. Basic information is being sought on the factors which govern the rate of extraction of a dissolved substance by the second liquid phase, in particular the kinds and extent of motion in the phases and, if one phase is present as droplets, the nature of movements within them. This information is being obtained by studying droplets in a hydraulic tunnel.

Surface films. A study has been made of the physical and chemical properties of spread monomolecular layers of compounds similar to those present in wool wax. It has been shown that the monolayer techniques may be used advantageously for determining the general architecture of molecules containing between 12 and 40 carbon atoms, and for determining the positions within the molecule of chemically reactive groups. It is considered that the techniques will be particularly useful in determining the structure of materials available only in small amounts, since only 0.0001 g. is required.

The structure of eburicoic acid, a triterpene occurring in certain wood fungi, has been determined and shown to be similar to lanosterol, a constituent of wool wax. The configurations of a number of other compounds have also been determined.

Attempts have been made to reduce evaporation from free water surfaces, a problem of some importance in areas of low rainfall, by covering the surface with insoluble surface films. A number of films were developed and found to be unaffected by wind and dust. Nevertheless these films failed generally to reduce the natural rate of evaporation of water. As a consequence the long-held view that the failure of surface films results from damage has been abandoned, and the lack of success has been traced to compensating effects resulting from heating of the water by the sun. Alternative methods of water conservation are being examined.

Wool wax recovery. The flotation method for recovering wool wax developed in the Physical Chemistry Section of the Division has been modified so that the wax produced is superior to that usually obtained by other methods. The process in its final form consists in aerating scour liquors in modified mineral flotation machines, recirculating the froth through the flotation machine impellers to reduce its bulk and then, after washing the froth in counterflow with water, dispersing it in hot alkaline solutions to give a concentrated emulsion from which wax is separated by centrifuging. The overall recovery of wax from the liquors is approximately 50% for liquors containing 1.5% wax and greater for richer liquors. One Australian scourer is already using the process successfully on a commercial scale and wide interest has been aroused both in Australia and overseas. The Geelong laboratory of the Wool Textile Research Labora-

tories is now co-operating with the Division of Industrial Chemistry in assisting interested firms to install commercial units.

Wool wax chemistry. The Organic Chemistry Section of the Division has continued its examination of the aliphatic alcohols occurring in combination in wool wax, with particular reference to the normal (straight-chain) alcohols. There has been some doubt in the past regarding the occurrence of such alcohols in wool wax; it is expected that the present work, using the techniques of chromatography and fractional distillation in the spinning-band column, will settle the question.

The acidic components isolated from the natural wax are also under study by reducing them to the corresponding alcohols, and applying to these the above techniques.

Catalytic hydrogenation procedures for converting the wool-wax acids to the commercially more useful alcohols have been studied. Small-scale hydrogenations under high pressure (4,000 p.s.i.) at temperatures of the order of 300°C. in the presence of copper-chromite catalysts have given good yields; it has also been found possible to reduce the copper soaps without further catalyst. Direct hydrogenolysis of wool wax itself under similar conditions has also been studied and found to proceed more readily than the reduction of the acids.

Various derivatives of the wool-wax acids have been prepared, but do not appear to have any immediate industrial application.

Some experiments directed towards the preparation of modified alkyd resins using wool-wax acids in formulations with phthalic anhydride and glycerol have yielded products which may find a place in the lacquer industry.

Flotation of cassiterite. Work on the flotation of cassiterite from the ore from Maranboy, Northern Territory, was suspended while still in its preliminary stages in order to complete the development of the process for recovering wool wax from scour liquors. Work on this ore has now been resumed and the results have been sufficiently encouraging to justify an intensive investigation.

Condensation of steam. The greatly improved heat-transfer coefficient of a condenser surface on which condensation occurs as droplets rather than a film has been further investigated by the use of an experimental boiler and condenser. A number of substances has been used to render the surface of the condenser hydrophobic, but so far their effect in causing vapours to condense in a drop-wise fashion has been only temporary. The range of surface treatments is being extended. Concurrently, a study is being made of the fundamental mechanism and kinetics of the process. A quartz microbalance is being made for the measurement of the adsorption of water on hydrophobic surfaces and is nearly complete. It will be of general value for adsorption studies and for the determination of surface areas of powders.

Heterogeneous catalysis. Because of the widespread use of catalysts to accelerate reactions in the manufacture of chemicals, an understanding of their functions is of considerable importance in achieving maximum yields at low temperatures. Work was concluded during the year on the rate of transport of gases and their reaction in the pores of two industrially important catalysts, thoria and alumina, and the way these rates are affected by pressure, temperature and size of pores. This work is described in a series of papers now in the press.

ORGANIC CHEMISTRY

The research programme has continued in the main directions described in the previous report and is chiefly directed to the study of the chemistry and utilisation of wool wax and sugar-cane wax, the examination of Australian plants for alkaloids of possible pharmaceutical value and of substances toxic to livestock, plastics investigations with particular reference to the mechanism of the phenol-formaldehyde reaction, and the examination of seed oils from Australian plants.

Sugar-cane wax

The systematic examination of derived waxes, prepared from the mixture of free fatty acids which results from the bleaching process, has been continued. These derived waxes are made by esterifying the carboxyl group with a polyhydric alcohol or by converting it to a diamide; the effect of such chemical modification on the physical properties has been studied, concentrating on the requirements for a solvent paste polish. The requirements of a hard wax for this use are exacting, so that if a derived wax could be made which satisfied them, it should be possible to make waxes from sugar-cane wax suitable for most industrial uses. The methylene and ethylene diamides and the pentaerythrityl ester waxes are the most promising of each group, and confer very high gloss on polishes made with them, while blends of the two groups are valuable in eliminating certain defects of each. The waxes mentioned compare very favourably with carnauba wax or its synthetic substitutes. It has also been shown that the differences in properties between bleached acidic waxes prepared with and without vacuum refining are reduced in their derivatives, and it appears possible that cheaper yet still useful derived waxes may be obtained by omitting the distillation under reduced pressure. A patent application covering the use of sugar-cane wax derivatives in polishes has been made. A patent covering earlier stages of the process has been obtained here and in the U.S.A.

Solvent refining of crude cane wax has been further studied, principally in regard to the use of acetone at pressures above atmospheric.

ORES AND METALS

Ore dressing

At Melbourne, where work is done in co-operation with the Department of Mining of the University, 30 reports were issued. These refer to the development of treatment methods for ores of gold, copper, lead, zinc, tungsten and tin and to work done on beach sands, magnetite, alluvial diamonds, antimony slag, brown coal overburden and brass dross.

Together with investigations not yet completed, these indicate continued interest in the treatment of tin and tungsten ores, and in the recovery of gold from sulphide concentrates produced at Victorian gold mines. There is also a revival of interest in cobalt ores.

Work was done on the application of gravity and magnetic methods of concentration to recover wolfram and scheelite from a fine-grained ore from southern New South Wales. The relative efficiencies of two flow sheets, each of which involved stage-grinding and concentration, were compared. An interesting feature of this ore is that not only the wolfram but also a large proportion of the gangue is magnetic.

Following the investigation of samples of magnetic ore, a plant has been erected in New South Wales to provide magnetite medium for heavy media coal cleaning plants.

Work reported in connection with beach sands has been to a large extent concerned with the production of high-grade monazite concentrates. Work, not yet reported, has been done on the elimination of chromite from an ilmenite concentrate by magnetic separation after heat treatment.

Investigation of an alluvial gravel from New South Wales showed that the diamonds were very poor conductors, while the gravel (mainly quartzite and tourmaline) was relatively conducting. The relatively high conductivity of the quartzite was unexpected. Electrostatic separation was possible at a size coarser than 6 mesh. The company concerned has since built a pilot roll-type separator to carry out tests in the field.

Work, not yet reported, has been done on the electrostatic separation of silica from granular lime-sand from Western Australian beaches. No appreciable separation was possible when the mixture was cold even after thorough drying. However, on heating to 90°C., the silica developed strong negative charges and the lime positive charges, which made possible a remarkably good separation. The Western Australian Department of Industrial Development has since built a pilot plate-type separator.

Investigation of the rate of adsorption of caprylic acid at a nickel-water interface indicated that the adsorption proceeded with no activation energy. The adsorption was reversible and

followed the Gibbs adsorption equation. Multilayers of acid were formed on the metal surface.

Further development of a polarographic method for the determination of oxygen in treatment-plant solutions has taken place. It is possible to determine the oxygen content of simple flotation and cyanide pulps quite readily with an accuracy better than 5%. Ions of Cl^-, SO_4^{2-}, CNS^-, $S_2O_3^{2-}$, $Fe(CN)_6^{4-}$ in concentrations normally met in practice do not interfere; neither does 0.1% Cu. In the presence of zinc the determination is possible but more difficult. Lead at low alkalinities has no effect, but at high alkalinities there is serious inteference with the method. Further work is proceeding.

Titanium and its alloys

Alloys. The chief aim of this work has been to provide a general understanding of the factors which affect the alloying properties of titanium. The study of titanium alloys containing elements with atomic sizes close to that of titanium has been continued and detailed investigations of titanium-tin and titanium-silver alloys have been completed. The results of these and earlier investigations by the Section have been considered, together with results from overseas laboratories, with a view to deducing empirical laws relating to the formation of solid solutions in the allotropes of titanium. It has been found that solutions of non-transitional elements in both forms of titanium seem to conform with rules relating to atomic size and crystallatice geometry. Solutions of the transitional metals in the high-temperature form of titanium also appear to obey these rules, but solutions in the low-temperature form do not.

Reaction with oxygen. The extremely rapid oxidation of titanium and its alloys at temperatures in excess of 500°C. is a major problem in the development of these materials for use at high temperatures. It has been found that the diffusion of oxygen in the titanium metal during the progress of oxidation largely governs the rate at which the oxidation proceeds. This is unusual because, in general, the oxidation of metals and alloys is almost invariably controlled by the diffusion of metal or oxygen through the intervening oxide layer.

An oxidation mechanism which is controlled by diffusion of oxygen within the metal itself suggests that the high-temperature oxidation of titanium may be reduced by suitable alloying of the metal. This aspect will be investigated. An *x*-ray diffration study of the lower oxides of titanium has been completed and the results have been used in interpreting the composition and structure of various oxide scale layers formed during the oxidation of titanium.

Chemical studies. The chemical compositions of the oxide scales formed on the surface of metallic titanium in the oxygen surface-reaction work have been investigated. It has been found that the presence of some metallic foreign elements can have an important effect on the oxidation rate. Chemical methods have been used to verify the oxygen distribution between the scale and the metal.

The reactions between titanium monoxide, sesqui-oxide and non-stoichiometric dioxide and chlorine have been investigated.

The thiocyanate-tungsten reaction is being investigated as a method for the estimation of tungsten in titanium metal.

British Paper and Pulp Mills in America

DETAILS OF BOWATER'S NEW TENNESSEE UNDERTAKING

As we noted in our September issue, the new paper and pulp mills recently brought into operation at Calhoun, Tennessee, represent the biggest investment of British capital since the war. Erected at a total cost of $60 million (some £20 million), the mills are operated by Bowaters Southern Paper Corporation, a wholly-owned subsidiary of the Bowater Paper Corporation Ltd., of London. The mills' two paper machines are stated to be the most modern of their kind and can produce a sheet of newsprint nearly 20-ft. wide at a rate of 2,000 ft./min. The initial design capacity is for the production of 130,000 tons of newsprint p.a. and 55,000 tons of either unbleached or bleached kraft pulp for sale.

The newsprint furnish requires approximately 360 tons/day of pine groundwood and 120 tons of bleached kraft pulp, with the total kraft pulp requirements, including that for sale, being some 312 tons/day.

The chemical consumption generally requires about 20 tons/day of salt cake, 8 tons/day of caustic, from 8 to 18 tons of chlorine and 14 tons of lime. For the news furnish about 4,000 gal. of 32° Baumé liquid alum is used, and 1,200 gal. for water treatment, with required quantities of limestone, soda ash and chemicals for slime prevention, colour, etc.

The plant will have between 700 and 750 employees and at least an equivalent number will be employed directly or by independent contractors for pulpwood production. Approximately $3½ million will be paid annually for wages at the plant, and a similar amount, or more, in connection with the wood supply.

Digesters. For the daily production of 312 tons of raw kraft, there are five digesters, 11 ft. 6 in. i.d. by 43 ft. 3 in., of 3,300 cu. ft. capacity, with space left in the building for a sixth. Each digester has a shell thickness of 2 in. and weighs approximately 62 tons. The digesters discharge through electrically operated blow valves through individual lines to a blow tank having a capacity for three blows.

The blow steam, after passing through a special cyclone in the head of the blow tank, goes to an accumulator and blow steam recovery system for the heating of the water.

The relief steam from the digesters is carried to individual separators, thence to a relief cyclone, condenser and decanter for the recovery of turpentine.

Bleach plant. From a screened stock chest in the wash-room basement, as much pulp as it is desired to bleach is pumped at regulated consistency through brushing jordans to a tonnage control unit serving the chlorinators of a three-stage bleaching plant.

The chlorination stage consists of a tile-lined steel tower, 18 ft. diameter by 61 ft. high, with two-tier arrangement of rubber-lined steel circulators and a rubber-covered skimmer to the launder at the top of the tower.

Chlorinated stock is pumped to an 8 by 14 ft. rubber-covered single-valve vacuum washer, with shredder and conveyor for discharging through the high-density double-shaft mixer of cast iron to a caustic extraction tower of unlined steel, 10 ft. diameter by 47 ft. high, with one circulator of cast iron and 304 stainless-steel construction.

Stock at the bottom of the caustic tower is diluted to about 2½% consistency by nozzles for pumping to a cast-iron single-valve vacuum washer with shredder and conveyor for delivery at high consistency through a double-shaft mixer to a steel, tile-lined hypochlorite tower, 17 ft. diameter by 17 ft. high, provided with two 304 stainless-steel circulators.

At the bottom of the hypochlorite tower, the high-density stock is diluted by nozzles and pumped to an 8 by 14 ft. final vacuum washer with rubber-covered repulper, this washer being arranged for use with a future SO_2 stage, should the bleach plant be expanded.

Stock from the final washer passes through a pan-type consistency regulator which discharges directly into an agitated low-density tile storage tank, 16 ft. diameter by 18 ft. high, from whence it can be pumped either to the newsprint mill or to the pulp drier, as desired.

While the immediate plant is a three-stage for semi-bleached, full provision has been made for adding another caustic, hypochlorite and SO_2 stage or for chlorine dioxide should the mill desire to manufacture full bleach.

Recovery. For the evaporation of the surplus black liquor from the kraft mill, a six-body, sextuple-effect evaporator has

been installed. The unit has sufficient capacity to evaporate 173,000 lb./hr. of water, with 34,500 lb./hr. of steam at 35-lb. gauge.

To condense the vapours from the sixth effect, a surface condenser of the vertical type is used, and consumes 1,800 gal./min. of 70° water, with a temperature rise to 110°. A soap skimming tank with a paddle-type mechanical skimmer removes the soap from the liquor between the third and fourth effects.

The chemical recovery unit for the burning and reducing of the dry solids contained in the black liquor has capacity sufficient to burn 950,000 lb. of solids per 24 hr., with a steaming capacity of 123,200 lb./hr. at 875 p.s.i. and 825° from the feedwater at 280°.

Preliminary evaporation on the unit is through a cascade evaporator, with wheels 14 ft. diameter by 9 ft. 6 in. face. Both f.d. and i.d. fans are driven by motors, the latter having a hydraulic coupling for speed control.

For soot blowing, an air puff system has been furnished for the collector zone and the economiser passes, consisting of 16 heads automatically controlled for sequential operation. In addition there are 12 retractable soot blowers for the clearing of the screen tubes and superheater elements, all arranged for double-side installation at six locations. These units are air operated with superheated steam for the cleaning medium.

For the handling of salt cake a pneumatic system is used to unload cars and deliver either into storage or the feed hopper of the unit.

For the recovery of salt cake and dust in the flue gases, an electric precipitator is furnished and installed as an integral part of the recovery building above the cascade evaporators. The unit consists of two electrical precipitators enclosed within a shell structure of combined hollow tile, steel and reinforced concrete. Each of these units has an individual stack and is so arranged that with one of the units down, the other can still be maintained in operation.

The recovered salt cake from the precipitator is collected and dissolved in four wet-bottom pans through which concentrated liquor is circulated and its chemical strength built up.

Causticising. The causticising plant consists of the following conventional systems: green liquor clarification, dregs washing, lime slaking, white liquor clarification and lime mud washing. The design capacity is 30,200 cu.ft./day of white liquor, with 8 lb./cu.ft. of total alkali and 7 lb./cu.ft. of active alkali, all expressed as Na_2O with a sulphidity of 30%. The plant can be operated up to a capacity of 38,000 cu.ft./day. The white liquor clarifier and lime mud washer are of the multiple-tray type, while the green liquor clarifier and dregs washer are of the single-tray type.

Lime reburning. The lime reburning plant consists of a rotary lime kiln, 9 ft. by 250 ft., with four supports. At the feed end, a vacuum mud filter and a slurry feeder provide a constant rate of feed. Fuel for the kiln is natural gas. The design capacity of the kiln is approximately 80 tons/day. The kiln can be operated up to a capacity of approximately 125 tons/day to handle peak loads and additional production. Limestone is used as makeup and is introduced by a table-type feeder to the kiln feed conveyor. The limestone is stored in an overhead storage bin with a capacity of 47 tons, and provision has been made for a ground storage of approximately 100 tons. The limestone system consists of a track hopper, drag chain conveyor, bucket elevator and necessary chutes.

Recent publications

Pinch valve. The Warren Morrison pinch valve, which, it is claimed, can handle corrosive liquors, abrasive slurries, delicate chemicals and foodstuffs as well as clear liquids or gases, is described in a two-page pamphlet. The valve consists of a natural or synthetic rubber tube, with end flanges to form the pipe joints, a clamping mechanism and a metal body. The tube may be regarded as an easily replaceable rubber lining and the employment of several grades of specially compounded rubber ensures efficient operation for considerable periods with a wide range of fluids. The body is so designed to give the maximum support to the rubber tube and to prevent more than slight seepage of liquid occurring if for any reason the rubber tube becomes fractured. Valve closure is effected by two anvils, which are brought together simultaneously on the centre line of the valve; stops are provided to prevent damage to the rubber tube by excess strangulation. When open, flow resistance is negligible. When throttling, fine control is possible down to a positive shut-off. Gland leaks and the problem of sticking are eliminated; the metallic working parts are isolated both from the fluid in the valve and from atmospheric conditions.

Automatic controls. Crosby Valve and Engineering Co. Ltd. have issued an 11-page catalogue on automatic controls. This deals with their various liquid level controllers as well as valves, regulators and associated equipment. The liquid level controllers are of two types, the variable displacement type and ball float type. The advantages of the variable displacement type are: (1) it may be used for level ranges as short as 14 in. and as long as 15 ft. or over; (2) it eliminates the necessity for a stuffing box; (3) it provides the accuracy and flexibility of instrument control. The instrument may be proportional or proportional-reset type, or it may incorporate two control assemblies, or a control assembly plus a pneumatic transmitter, or a transmitter or a pneumatic switch only. They can be mounted on equipment such as vessels or tanks, internally or externally. In the ball float types the movement of the counterweight-balanced float is a direct indication of level changes. It is useful in many short-range applications. A wide selection of control valves and pilot-operated equipment is also illustrated.

Selenium rectifiers, for a.c. and d.c. conversion for electroplating, the operation of electrolytic cells, the charging of batteries and other applications where heavy currents at low voltages are required are described in an illustrated leaflet from Hackbridge & Hewittic Electric Co. Ltd. A further application is electrostatic dust precipitation where very low currents at very high voltages are required.

Whilst for nominal outputs the rectifiers are naturally air-cooled, for large outputs the company produces units both of the naturally oil-cooled and of the forced-air-cooled types.

Whereas the naturally-oil-cooled type is completely static and protected against corrosive atmosphere, the forced-air-cooled type offers advantages where operation in a non-corrosive atmosphere is required, in that it enables substantial saving in cost, weight and space.

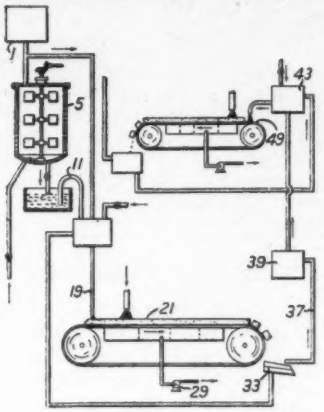
Industrial radiography materials. The requirements of industrial radiology are extremely varied and can only be met adequately by a comprehensive range of X-ray films and screens. For example, the critical X-ray examination of magnesium and aluminium alloys demand a fine-grained high-contrast emulsion suitable for direct exposure to X-rays, but thick steel welds generally require a combination consisting of a faster type of X-ray film and either lead screens or salt screens. Ilford Ltd. have recently issued a 20-page illustrated catalogue giving details of the X-ray films, screens and chemicals they supply, with instructions for their correct use.

Noise and vibration measurement. This 34-p. pamphlet published by A. E. Cawkell, electronic engineers, follows a previously produced pamphlet, 'Noise and Vibration in Industry,' in which were discussed fundamental definitions, methods and various difficulties encountered. This second paper discusses the subject in greater detail and sets out experiences gained in the past few years, particularly with mechanical test rigs and electronic measuring apparatus. Noise measurement sets a number of problems which need to be individually considered, general principles are therefore discussed without reference to particular practical applications. The booklet concludes with a detailed consideration of the electronic equipment necessary for this type of work, and includes descriptions of power supplies, amplifiers, meters, oscilloscopes, filters and various other auxiliaries, with photographs of instruments and factory test rigs.

British Patent Claims

Producing sintered iron catalysts

Iron catalysts for use in the catalytic conversion of carbon monoxide and hydrogen are obtained by subjecting iron oxide mixed with a small amount such as 2 to 8% of carbon, and from 0.5 to 10% of conventional promoters such as alkali metal



compounds or the oxides of chromium, zinc, magnesium, manganese or rare earth metals, to combustion and sintering in an oxidising atmosphere at 2,200° to 2,500°F., preferably followed by reduction in hydrogen at 600 to 1,100°F.

The iron oxide may advantageously comprise sintered pyrites ash, from 2 to 15% by weight of oxides of antimony, vanadium, manganese or copper or 25 to 30% of aluminium oxide being incorporated prior to sintering with carbon.

In the figure, pyrites concentrate from bin 1 is roasted at 700 to 1,200°F. in furnace 5 to reduce the sulphur content and passed *via* lines 11 and 18 to a moving grate 21 and sintered at 2,000 to 2,300°F. in an oxidising gas drawn downwardly through the mass by suction pump 29, thus combusting the residual sulphur and sintering the oxide mass. The latter, collected at 33, passes *via* 37 to a crusher 39 and hence to a mixer 43 where 5% by weight of coke and 5 to 10% of water which may contain 0.5 to 3% of potassium carbonate or fluoride, are added. The mixture is then subjected to combustion of carbon and consequent sintering on the moving grate 49, any portion of the layer being exposed to sintering temperature for about 1 min. The oxidising gas is drawn downwardly through the mass as before. The product is crushed to 30 to 200 microns size and reduced in conventional manner.—658,848, *Standard Oil Development Co.*

Houdry catalyst regeneration system

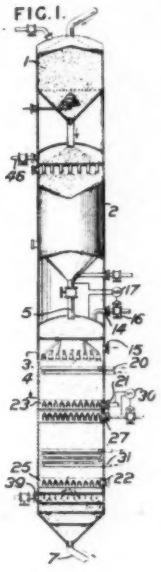
Hydrocarbon conversion catalyst is regenerated by moving it downwardly as a compact bed in countercurrent to an oxygen-contained regenerating gas through a plurality of combustion zones. The regenerating gas and the gaseous combustion product are separately supplied to and withdrawn from each zone so as to prevent admixture of the gases in adjacent zones. Increased gas pressure obtains in each succeeding combustion zone and the combustion products are discharged from one zone at a pressure substantially equal to that of the regenerating gas entering the immediately preceding zone.

As shown, catalyst is treated with steam in hopper 1, then falls through pipe 4 to hydrocarbon conversion zone 2 where it moves in concurrent or countercurrent direction to the reactant vapours, and finally falls through pipe 5 to the regenerator 3 which it leaves at 7 and is returned in a closed system or otherwise by an elevator to the hopper 1.

The regenerator is divided into two separate combustion zones in the lower of which regenerating gas enters at 22 through distributor 25 and combustion products leave at 27, and in the upper of which regenerating gas enters at 21 through distributor 23 and combustion products leave at 15. Cooling coils 20, 31 are supplied in the respective zones.

Seal gas is fed to the top of the regenerator at 14 and to the top of the reactor at 46

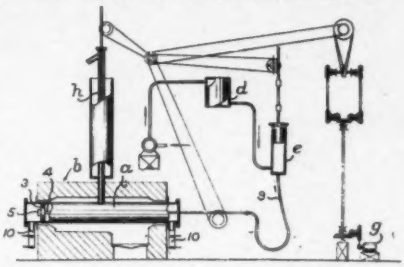
FIG. 1.



and the difference of pressure between the foot of the elevator and the discharge outlet of the regenerator may be made up by admission of pressure gas through line 39. Differential pressure control means 17, 30 are provided to prevent admixture of gases between the reactor and regenerator and between the different zones of the regenerator.—667,584, *Houdry Process Corporation.*

Cracking vegetable tar

Vegetable tar is cracked by applying a moving spray of the liquid tar against the inner wall of a container heated to a temperature between 350° and 500°C.; and the vaporous products so formed are introduced into a fractionating column from which various condensates may be withdrawn. These condensates are suitable as solvents or, when mixed with ethyl alcohol, as fuels in internal combustion engines.



In the diagram, the container comprises a metal cylinder *a* which is heated to about 500°C. by furnace *b*; and the tar is sprayed against the walls of the cylinder through atomiser 5 which forms part of the working-face of reciprocating piston 3. The tar is supplied from reservoir *d*, from which it is pumped by pump *e* to atomiser 5 *via* flexible hose 9 and the bore of hollow piston-rod 4.

The vapours, formed by the cracking, are passed to rectifying column *h*; and the carbon-residues are discharged into two sumps 10. The light fractions are mainly composed of benzene, toluene, xylene, cumene, chrysene, and saturated acyclic hydrocarbons; and it is possible to convert 28% of the tar into hydrocarbons boiling between 70° and 200° C.—670,479, *F. M. Mora.*

Fibrous polytetrafluoroethylene

Orientated and fibrous polytetrafluoroethylene is obtained by passing micropulverised, pulverised, granular or lump polytetrafluoroethylene through milling rolls under pressure. The polytetrafluoroethylene treated may be scrap obtained in the fabrication of sintered articles and may be mechanically subdivided prior to mixing. After milling, the fibrous material may be shredded, *e.g.* mechanically or by beating a slurry in a liquid medium, *e.g.* water and/or organic liquids such as ethanol, removing the liquid and drying. Prior to, or after milling, *e.g.* in the slurry, binding materials, *e.g.* silicon resins, polyvinyl butyral and/or lubricants, *e.g.* graphite, petrolatum, calcium stearate, modifying agents, *e.g.* dibutyl sebacate, and fillers, *e.g.* asbestos, may be mixed with the polytetrafluoroethylene. The fibrous material may be braided, made into string or used as packing or gasket material. —642,269, *E. I. Du Pont De Nemours & Co.*

World News

GREAT BRITAIN

Mond—Birlec—A.E.I. agreement

The Mond Nickel Co. Ltd., on behalf of its parent company, the International Nickel Co. of Canada Ltd., announces that following negotiations between Associated Electrical Industries Ltd., Mond and Birlec Ltd., the sale by Mond of its wholly owned subsidiary, Birlec, to A.E.I. has been agreed.

Birlec will operate as an individual company within the A.E.I. Group. Mr. G. P. Tinker will continue as managing director and Mr. T. G. Tanner and Mr. J. H. Crossley as executive directors.

A 55-ft. distillation column

A 55 ft. long, 2 ft. 6 in. diam. distillation column, containing 40 Shell-patented *Turbogrid* distillation trays, was recently despatched by road by Metal Propellers Ltd., who are now manufacturing these trays under licence.

The column is the longest complete plant ever despatched in one piece by the company. Both column and trays are manufactured from Monel. The column was loaded on a 40-ft.-long, six-wheeled trailer and taken to the Shell oil refinery at Stanlow, Cheshire.

Epoxide resins patent agreement

An agreement has been reached between Aero Research Ltd. and Shell Chemicals Ltd. covering, in the U.K., the patents held by Ciba and Shell in the field of epoxide resins.

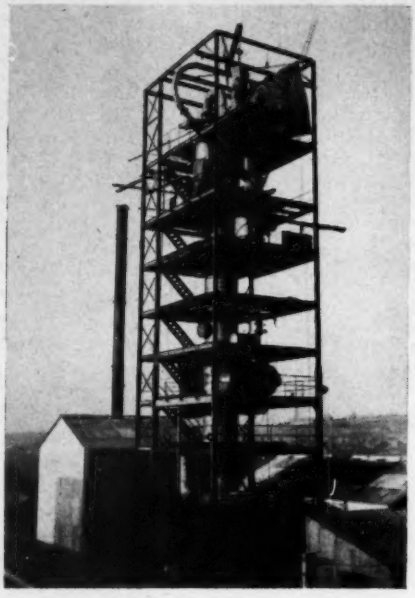
This agreement will be of particular value to the paint industry in that it will give freedom under both the Ciba and Shell patents to all purchasers of epoxide resins from either company to sell or use these materials in the surface coating field.

In the specialised fields of potting, casting, laminating and adhesives, similar freedom under the patents will be available to customers purchasing epoxide resins from either company for their own use but not for resale in their original or modified form.

Oil refinery's expansion

The post-war expansion scheme at Anglo-Iranian's Grangemouth refinery, on the Firth of Forth, was recently completed with the commissioning of the last three units—a 60,000-tons p.a. catalytic polymerisation unit, a *Girbotol* unit and a sulphur recovery unit. The scheme has raised crude oil capacity from 360,000 tons p.a. immediately after the war to 2,700,000 tons today.

The sulphur recovery unit will initially produce about 14 tons/day of sulphur. Gas from the cracking plant first passes through the *Girbotol* unit, which removes its sulphur content. Then it is fed into the catalytic polymerisation unit and processed into two products—high-octane motor spirit and tetramer, which is piped to the British Petroleum Chemicals Ltd. factory adjoining the refinery for use in the manufacture of chemicals.



Main distillation structure of the Littleborough, Lancashire, plant where Hess Products Ltd. produce high-purity fatty acids using the process devised by Armour & Co. of Chicago. Hess Products new plant for making a new range of chemicals, as described on another page, is to be erected on an adjacent site. The buildings seen in this photograph are, *left to right*, the Dowtherm boiler house, pump house and control room at the present plant.

The sulphur extracted by the *Girbotol* unit is processed by the recovery unit into flake sulphur. This sulphur is transported by road to Broxburn oil works, where it is manufactured into sulphuric acid for use in the chemical industry.

Propane and butane project

Now under construction at Shell Haven oil refinery, Essex, and expected to be completed by mid-1955 at a cost of £300,000 is a new plant which will produce and handle 10,000 tons p.a. of butane and propane. In the U.K., consumption of these products has increased by several hundred per cent. since the war and it is estimated that, apart from industrial concerns, which use them for metal cutting, brazing, paint scaling and other operations, well over 1¼ million people are dependent on them for cooking, heating or lighting.

To keep the gases liquid at normal temperatures it is necessary to store them under pressure. At Shell Haven the new storage facilities will consist of large spherical tanks for butane and ' bullets '—high-pressure, long, cylindrical vessels—for propane.

A bottling plant is also planned. This will contain equipment for pressure-testing the steel bottles and bottle-filling machines. For the time being only propane is to be bottled, but bulk loading facilities are also to be installed for filling road tankers and rail cars with both propane and butane.

Constructional work has already begun and the site is being prepared by John Laing & Son Ltd.

Chemical and Petroleum Engineering Exhibition

Arrangements have now been completed to hold for the first time in the U.K. a joint exhibition of chemical plant and petroleum equipment. This exhibition will be known as the Chemical and Petroleum Engineering Exhibition and will take place in the Grand Hall, Olympia, London, in June 1958. It will be organised by F. W. Bridges & Sons Ltd. and sponsored jointly by the British Chemical Plant Manufacturers Association and the Council of British Manufacturers of Petroleum Equipment.

Atomic power engineering school

As a step towards encouraging industry to play a greater part in the development of atomic power, the United Kingdom Atomic Energy Authority is opening a reactor school at Harwell. The new school will provide, for a fee of £250, a three-months' course of training for staff from industrial concerns to learn the techniques by which heat from atomic piles can be converted into useful power.

Three courses will be held each year starting in September, January and May, each course lasting three months. The syllabus includes lectures and individual instruction in nuclear physics, reactor physics, metallurgy and reactor engineering, as well as experimental work. Lectures are given by experts drawn from various divisions at Harwell, and by the full-time school staff.

The school is at present in temporary accommodation, which comprises lecture hall, office, laboratory and counting room. About 25 students can be accommodated.

Among firms with students at the school at present are Associated Electrical Industries, the British Electricity Authority, Babcock & Wilcox, English Electric, General Electric, Parsons, Rolls-Royce, John Thompson, and Whessoe.

Welding research display

The metallurgical laboratories of the British Welding Research Association are holding ' open days ' on November 23 and 24, 1954, at 29 Park Crescent, London, W.1. Researches on both ferrous and non-ferrous alloys will be displayed covering many welding processes including the argon arc, metal arc and self-adjusting arc processes. Tickets are available from the secretary of the Association.

Change of telephone number

The telephone number of British Filters Ltd. has been changed to Maidenhead 4144.

Non-ferrous foundries amalgamation

Two Stoke-on-Trent non-ferrous foundries, T. M. Birkett & Sons Ltd. and Billington & Newton Ltd., have amalgamated. The company will be known as T. M. Birkett, Billington & Newton Ltd., and will be one of the largest non-ferrous founding firms in the country. The board of directors remains unchanged: A. P. Wright (managing director), J. C. M. Sheckell, A. Birkett, E. Y. Birkett and F. Bennet. Headquarters of the company will be a newly erected administrative block at Hanley which includes spacious office accommodation, drawing offices and a laboratory. The foundries and machine shops at both Hanley and Longport will remain in operation.

Birkett, Billington & Newton make castings weighing from a few ounces to 10 tons in a variety of non-ferrous metals including phosphor bronze, gunmetal, aluminium bronze, manganese bronze and light alloys.

Greases and compounds

Acheson Colloids Ltd. announce that the demand for *Gredag* greases and compounds has made it necessary to establish a division to handle the growing volume of business. This will operate from the company headquarters in London.

Another division was established, with its own factory, in 1953, to manufacture dispersions of finely divided pigments in all media as part of a co-ordinated plan of Acheson Industries Inc., of New York, of which Acheson Colloids Ltd. is a unit.

Quickfit's progress

Receipt of its first bulk order from the United States, and selection of the firm's new catalogue as one of the best of the year by the Federation of Master Printers and the Council of Industrial Design, were two successes of Quickfit & Quartz Ltd. referred to at the annual general meeting of the Triplex group of companies by Sir Graham Cunningham, chairman and managing director. He said that the company was beginning to reap the advantage of years of work by the technical research department, which had developed many new devices for improving output and speeding up production.

He also said that Q.V.F. Ltd., the new subsidiary of Quickfit which markets 'visible flow' industrial glassware, had got off to a good start.

Electronics exhibition

The Scientific Instrument Manufacturers' Association announces an exhibition with the title 'Electronics at Work' to be held at the Chamber of Commerce Hall, New Street, Birmingham, from November 23 to 25. The exhibits will include many instruments of interest to the chemical and allied industries.

Flexible tube and hose centre

To meet demands made on their 'flexible' advisory service in the Midlands and north of England, Compoflex Co. Ltd. have opened a flexible centre at their factory in Huddersfield Road, Oldham.

The centre is staffed by a team of specialists able to give advice on the most suitable flexible tube or hose to use in any given situation. A development section has also been started in Oldham to deal with the design of special flexibles for particular projects.

The Leonard Hill Technical Group—November

Manufacturing Chemist—Technology in the Fine Chemicals Industry, 4—Materials of Construction; Cost Control in the Chemical Industry, 5; Naturally Occurring Chlorine-containing Organic Substances; Preparation of Diacetyl; Chelating Agents; Progress Reports on Pest Control, Chemotherapy, Perfumery and Antibiotics.

Corrosion Technology—Detection of Corrosion in Chemical Plant; Metallic Corrosion in the Baking Industry; Wooden Vats; Corrosion in Hospital Practice; Lanolin Rust Preventives; Corrosion in the Motor-Car, 2—The Cooling System.

Paint Manufacture—Accelerated Weathering of Paints; Physics in Paint Manufacture; Protective Coatings for Tins; The Specific Surfaces of Fine Powders; The Fire Hazards of Painted Surfaces.

Atomics—Gamma Radiography in Oil Storage Installations; The Uses of Radioactive Isotopes in Tracing Sewage Flow; Radioisotopes in Industry.

Food Manufacture—Chocolate Biscuits—Three Million a Day; Radiation Pasteurisation of Fresh Fruit; Alaska Salmon Canning; Seaweed Harvesting and Processing Research.

Fibres (Natural and Synthetic)—Fibre Data Sheet—Synthetic Fibres; Electron Microscope; Man-made Fibres in the Rubber Industry; Wet Processing—A.A.T.C.C. Convention; Conference on Fibre Friction; New Condensation Polymers.

Petroleum—Azeotropic and Extractive Distillation, 2; Photography in Oil Research, 2; Plant Calculations for Petroleum Technologists, 1; Fuel Research, 1953; Corrosion in the Petroleum Industry, 1.

Building Materials Digest—Timber Identification for Builders, 4; New Kidbrooke School; Cornish Slate.

Muck Shifter—Public Works and Municipal Services Exhibition; Hydro-electric Development in the North of Scotland.

World Crops—Official Control of Husbandry; The Cotton Industry of India; The Development of Spraying Machinery; Cobalt—Deficiency in Pasture.

New H.Q. for Junior Institution of Engineers

The new headquarters of the Junior Institution of Engineers at 14 Rochester Row, London, S.W.1, was opened recently. The Institution claims to be the tenth in order of seniority, by virtue of its 70 years, among the professional engineering and associated societies having their headquarters in London and to be the only one which, covering all branches of engineering and its allied sciences, offers to the young man, still in the process of acquiring experience and professional qualifications, a participation in its affairs on absolute equality with senior members, whatever their professional status.

PORTUGAL

Nitrogen project

Plans to expand the capacity of the Portuguese nitrogen industry from about 13,400 metric tons to 52,575 tons have been announced. Expansion to this extent is expected to be sufficient to meet Portugal's increasing fertiliser requirements and to eliminate the need for imports.

The programme calls for construction of a nitric acid and nitrate plant—the first in Portugal—as well as for expansion of existing facilities. A total expenditure of 700,000 contos will be required to carry out these plans. The programme is expected to receive sufficiently high priority from the Government to ensure completion within the next three years.

Nitrogenous fertilisers now produced consist only of ammonium sulphate and calcium cyanamide. Fertiliser nitrogen consumption in Portugal in 1953 amounted to approximately 36,000 tons, predominantly in the form of ammonium sulphate. Agricultural experts believe that it would be desirable to use more nitrates on Portuguese soils.

TURKEY

Nicotine sulphate project

A factory is to be built in Istanbul to manufacture nicotine sulphate. It is reported that the necessary equipment has been ordered from Western Germany.

NORWAY

Fertiliser plant

At Glomfjord, north Norway, the Norsk Hydro chemical concern is building a £2½-million plant to produce 100,000 tons of complete fertiliser and 35,000 tons of nitrate of lime a year.

The plant, to employ 100 men, will be ready in summer, 1955.

ITALY

DDT plant

The Italian chemical firm of Montecatini is to supply Persia with a plant for the production of DDT worth more than $13 million. Persia is to pay for the plant with shipments of crude oil.

CHILE

Sulphur-refining technique

A new sulphur-refining technique which, it is claimed, would permit the processing of very low-grade ores is being considered by the Chilean Minister of Mines. Experiments were conducted by Dr. Francesco Roma, of the Italian National Association for the Control of Fuels and Bologna University, and a pilot plant using Chilean sulphur ores is to be put into operation shortly to assess the economic possibilities.

It is reported that full-scale installation of the necessary equipment would be

rather costly, but the competitive position of Chilean sulphur in the international market would be greatly improved. Deposits at present considered unworkable because of their low sulphur content could be utilised and a 90% extraction rate could be expected.

GERMANY

New oil refinery near completion

The Ruhrchemie A.G. expect their large new crude-oil-refining plant at Oberhausen to be completed soon. The plant, which will include parts of a former Fischer-Tropsch hydrogenation installation, will get crude oil from the Near East. There will be a catalytic cracking plant, using a processing method developed by the Socony Vacuum Oil Co., of New York. At the start about 280,000 tons p.a. of crude oil would be processed.

Production is expected to begin next spring, with an initial output of 280,000 tons p.a.

SPAIN

Potash deposit

Drilling at Astrain near Pamplona has revealed the existence of a potash deposit extending over an area of 52 sq. km. The potential yield of the deposit is provisionally estimated at 80 million tons of pure potassium oxide.

BRAZIL

Steel production plans

There are plans to build two new steel mills. The Jafet Group announced that its subsidiary, the Mineracao Geral do Brasil, which already operates the modern seamless tube steel mill at Mogi das Cruzes (Sao Paulo), had bought land at Conselheiro Lafaiete (Minas Gerais) on which to set up a semi-electrically operated steel mill to produce ferrous alloys and special steels. This new mill may be in operation in three to four years' time with an initial capacity of 120,000 tons p.a.

It has also been announced by the Cia. Siderurgica Paulista (COSIPA) that plans for their steel mill project at Piassaguera, near Santos, are almost complete. The initial production planned will be from 200,000 to 300,000 tons p.a. of steel, mainly of sheet steel more than 150-cm. wide, with the possibility of production being stepped up to 1 million tons.

AFGHANISTAN

Sulphur deposits

Sulphur has been found in some quantity in gypsum in the mountains immediately south of Mazir-i-Sharif. Sample shipments amounting to some 200 tons have been sent to Russia for assay and treatment. The Russians are showing considerable interest in this project, which the Afghan Government hopes to develop. It is rumoured that if the assay comes up to expectations the Russians will attempt to obtain concessionary rights.

★ PERSONAL PARAGRAPHS ★

★ Davey, Paxman & Co. Ltd. announce the following additions to their board of directors:

Mr. L. L. Bott, who joined Ruston & Hornsby Ltd. in 1944 as production engineer. In 1951 he was appointed general works manager of Davey Paxman, which included responsibility for the Boiler and General Engineering Division.

Mr. A. G. Howe, who in 1940 was appointed chief diesel engineer responsible for design, development and research and more recently was appointed chief engineer, covering the development of all the company's products.

Mr. T. L. Kendall, M.B.E., sales director of Ruston & Hornsby Ltd. He is a member of the Diesel Engine Users' Association and chairman of the Fuel and Lubricants Panel of the British Internal Combustion Engine Research Association.

★ Owing to pressure of business commitments, **Sir Kenneth Hague,** deputy chairman and managing director of Babcock & Wilcox Ltd., has been compelled to resign his chairmanship of their associated company, Edwin Danks & Co. (Oldbury) Ltd. **General Sir Kenneth Crawford,** K.C.B., M.C., has been appointed a director of Edwin Danks and chairman in succession to Sir Kenneth Hague. Following a distinguished career in the Royal Engineers, General Crawford was Deputy Chief of the Imperial General Staff from 1947 to 1949 and Controller of Supplies (Munitions) in the Ministry of Supply until 1953.

★ **Mr. Brian H. Turpin,** managing director of Q.V.F. Ltd., of Stone, Staffordshire, has recently returned from a visit to the U.S.A. and Canada, where he visited a number of installations of the company's products.

★ **Sir George E. Bailey,** C.B.E., chairman, and **Sir Felix J. C. Pole,** deputy chairman, have resigned from the board of directors of Metropolitan-Vickers Electrical Co. Ltd. The Rt. Hon. the **Viscount Chandos** (formerly Mr. Oliver Lyttelton), P.C., D.S.O., M.C., was elected a director and chairman of the board.

★ **Mr. I. M. O. Hutchison,** sales director of the Balfour group of companies, has returned to the U.K. from a six months' round-the-world air tour which took him to the United States, Canada, New Zealand, Australia, Tasmania and South Africa. He states that there are 'tremendous opportunities' for business in chemical engineering plant in Australia. While in Australia he made arrangements to start George Scott (Australia) Ltd., forming this new company in Sydney.

★ **Mr. J. W. Urban,** director of overseas relations, has been appointed a director of Monsanto Chemicals Ltd. He first joined Monsanto in 1929 and has had 35 years' experience in the chemical industry. He will continue his duties as director of overseas relations, in which he is responsible for personal contact with the company's agents, associates and other industrial interests in overseas markets.

The resignation of **Dr. W. D. Scott** from the Monsanto board has been accepted with regret.

★ **Mr. B. W. Galvin Wright** has been appointed publicity controller of Imperial Chemical Industries Ltd. in succession to **Mr. Sidney Rogerson,** who leaves the company's service on December 31, 1954.

★ **Mr. H. S. Tasker** has been elected a Fellow of the Institute of Metals, in recognition of his distinguished services to the Institute. The total number of Fellows is limited to 12. Mr. Tasker is chairman of British Titan Products Co. Ltd. and was a vice-president of the British Non-Ferrous Metals Research Association from 1935 to 1951.

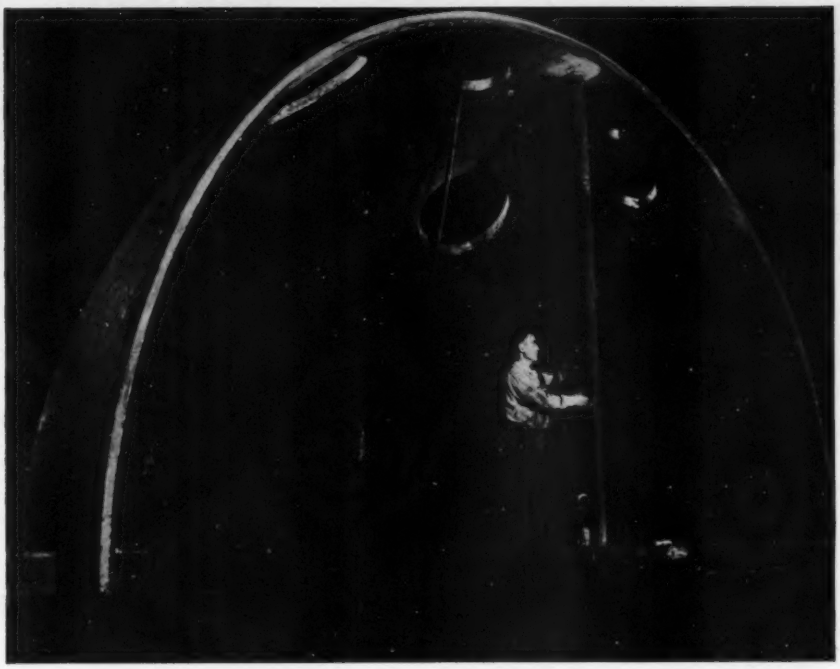
★ The following have been appointed members of the Advisory Council for Scientific and Industrial Research:

Mr. E. Ashby, president and vice-chancellor of Queen's University, Belfast; **Mr. S. Barratt,** finance director of Albright & Wilson Ltd.; **Prof. E. A. G. Robinson,** C.M.G., O.B.E., professor of economics, Cambridge University; and **Mr. C. Sykes,** managing director, Thomas Firth & John Brown Ltd.

Prof. R. S. Edwards, Lord Halsbury, Sir Henry Hinchliffe and **Mr. A. H. Wilson** retire from the Council.

★ **Mr. G. G. Buckingham** has been appointed manager of the Manchester office of Babcock & Wilcox Ltd. in succession to the late Mr. F. W. Woodfield, whose death was announced recently. He was appointed to the Manchester office of Babcock & Wilcox Ltd. in February of last year, following upon the absorption of the Stirling Boiler Co. Ltd.

★ **Mr. W. Roy Geddes,** vice-president of North American Cyanamid Ltd., died recently at the age of 57. He had been associated with the company, a subsidiary of American Cyanamid Co., since 1948, when he was named assistant to the president. He was elected vice-president in January 1953. His war-time services took him to London and Washington, as well as to Ottawa. He was Canadian civilian member of the Combined Production and Resources Board. He was awarded the C.B.E. for his war-time services. In addition to his position with North American Cyanamid, Mr. Geddes was a director and president of Chemical Construction (Inter-American) Ltd., also a subsidiary of American Cyanamid Co.



Half the head of the huge process vessel described on this page. Here, in Kellogg's shops before shipment, a vertical strut pipe is being fitted to prevent distortion during transit.

UNITED STATES

World's heaviest process vessel

Loaded on flat cars, one-half to a car, the bottom head for what is claimed to be the heaviest process vessel ever designed recently left the Jersey City shops of the M. W. Kellogg Co. for its destination in Indiana. Each half of the head consists of four 'orange-peel' sections of carbon-moly steel plate, 2¾-in. thick, cut to exact size and welded together.

At the job site the halves are being joined to form the bottom of the reactor vessel for a 30,000-bbl./day fluid hydroforming plant now under construction by Kellogg. The reactor vessel will be approximately 135-ft. high. On top of this will stand a smaller vessel in which catalyst for the reforming process is regenerated. When completed, the unit will tower more than 200 ft. above its supporting pedestal.

PVC production

Scientific Design Co. Inc. reports successful initial operation of a new polyvinyl chloride resin plant constructed by one of Europe's largest chemical manufacturers. They are employing a suspension process developed by Scientific Design in collaboration with the European firm. The suspension process is stated to be an advance on the emulsion technique of PVC production, and it is claimed that suspension PVC has faster reaction and drying times, fewer impurities and is in general less costly to manufacture than emulsion PVC.

Development of a series of 'package' process designs for streamlined low-budget PVC plants with production capacities in the 4- to 15-million-lb.-p.a. range is also reported. The company believes that a strong market for package PVC plants exists among the country's nearly 500 smaller processors of PVC resin who are interested in manufacturing their own raw material to individual specifications.

Three new fluid catalysts

Three new grades of *Aerocat MS* microspheroidal fluid cracking catalyst have been developed by American Cyanamid Co.

Grade 50/60 (light) has been designed especially for use in fluid cracking units which operate at low velocities and are equipped with exceptionally good catalyst recovery facilities.

Grade 60/70 (intermediate) is designed for units operating at moderate to low velocities with 'average' recovery equipment.

Grade 75/85 (narrow) is designed for units which operate at highest velocities and have limited recovery equipment.

A wide variety of fluid cracking facilities has been coming into operation during the past two decades or more, arising out of the petroleum industry's continuing search for improvement in the methods of producing high-octane gasoline and other products. American Cyanamid carried out a study which showed that the varying operating conditions which have been thereby created could be classified into three broad types according to catalyst requirements, and intensive experimentation resulted in the new grades.

More 'Teflon' to be made

The plant for making *Teflon* (tetrafluorethylene) resin at du Pont's Washington works near Parkersburg, West Virginia, is to be enlarged to about double the present capacity. Construction began in the summer at the site, where the company initiated the first commercial-scale output of *Teflon* in 1950.

New ammonia plants

A new ammonia plant being built at Natrium, West Virginia, for the Columbia-Southern Chemical Corporation, is expected to be in production by April 1955. It is estimated that the plant will cost 3 million dollars.

The opening of the plant will mark Columbia-Southern's entry into the ammonia production field.

The Standard Oil Co. (Indiana) and the Sinclair Refining Co. are to build a large new ammonia plant in Hammond, Indiana, near two of their refineries. Hydrogen and other gases produced as by-products from these refineries will form the raw materials for the new plant. Output will reach 300 tons/day of anhydrous ammonia.

Tall oil research

The Tall Oil Association has established a contest to recognise outstanding unpublished papers and research work of persons working on tall oil products. The three best papers will be given awards of $500, $250 and $100, respectively.

Ammonia's 2,000 uses

The American chemical industry is confident that there will be plenty of ammonia on hand by 1957 and the U.S. Government's production capacity target of 3½ million tons p.a. appears to be a practical one. In a historical survey of American ammonia production which the Manufacturing Chemists' Association published in a recent issue of its journal, *Chemical News*, it is pointed out that ammonia today has some 2,000 industrial uses, including refrigeration, hardening of metals, manufacture of nylon, plastics and fertilisers, refining of petroleum products, destruction of water bacteria, flameproofing of fabrics, making of dyes, nitric acid, and a multitude of other essential processes.

Prior to World War 1 ammonia was a little prized by-product of the coking of coal. The threat of conflict, however, led to the discovery of a way to make it synthetically. The principles, still followed today, were first incorporated into the Haber-Bosch process. That process is based upon the formation of ammonia from the reaction of nitrogen and hydrogen under high pressures and temperatures in the presence of a catalyst. From the first successful American ammonia synthesis unit in Syracuse, New York, evolved an industry indispensable to the national welfare.

Since its birth in 1921, synthetic ammonia manufacture has been rapidly expanded, but the cycle of expansion really

got under way early in the World War 2 period. Plants now operating or under construction can produce 2.5 million tons a year. The 48 plants located in 22 states obtain about 10% of their hydrogen from coke ovens and about 90% from synthetic sources.

Possibly the most significant development in synthetic ammonia use in recent years has been in the manufacture of highly concentrated fertilisers. During the war the need for such products vied with the military's demand for ammonia-derived munitions. But since 1945 more and more ammonia has been used for fertilisers. A practice which is being increasingly followed on farms in the South and mid-West is the application of gaseous ammonia, under pressure, directly into the soil.

In 1953 agriculture consumed 72% of all the ammonia output; industry and military requirements took the balance.

CANADA

Electrolytic cobalt production

As a result of improvements in refining techniques, International Nickel Co. of Canada Ltd. has initiated production of electrolytic cobalt at its Port Colborne refinery. This marks the first commercial production of electrolytic cobalt in Canada.

Hitherto, Inco's entire cobalt output has been marketed as oxides and salts produced at its Clydach, Wales, refinery. Through its new process the company makes available a cobalt which will be particularly valuable in the manufacture of alloys where high purity is advantageous.

Cobalt is one of 13 elements recovered from the company's Sudbury District, Ontario, ores. Nickel, of course, is the principal product; the others are copper, platinum, palladium, ruthenium, iridium, rhodium, gold, silver, selenium and tellurium. In addition, Inco furnishes high-quality smelter gas for conversion into liquid sulphur dioxide and normal smelter gas for sulphuric acid production. Soon, iron will be recovered as premium-grade iron ore.

Sulphite plant

Operations are starting at a new plant near Welland, Ontario, which will produce elemental sulphur, sulphur dioxide and high-grade iron sinter from pyrites. Built by the Noranda Mine Co., it will have a rate capacity of approximately 350 tons/day of pyrites concentrate, and is expected to produce about 54,000 tons of sulphite p.a. Of this total, about one-third will be in the form of elemental sulphur, while the balance will be sulphur dioxide gas, which will be used by the adjoining North American Cyanamid plant in the manufacture of sulphuric acid.

Raw material for the new plant will come from the Noranda mine in Quebec.

MEETINGS

Chemical Society

November 18. 'Molecular Rearrangement,' by Prof. M. J. S. Dewar, 7.30 p.m., Imperial College of Science and Technology, London, S.W.7.

November 19. 'The Structure and Properties of Olefin Complexes,' by Dr. J. Chatt, 4.30 p.m., Chemistry Department, The University, Birmingham. Joint meeting with the Birmingham University Chemical Society.

November 19. 'The Chemical Nature of Some Metal Alkyls,' by Prof. G. E. Coates, 7.15 p.m., Royal Technical College, Glasgow.

November 26. 'Some Applications of Electron Diffraction to Problems in Inorganic Chemistry,' by Dr. L. E. Sutton, 5 p.m., Washington Singer Laboratories, Exeter.

December 3. 'The Polymerisation of Vinyl Ethers,' by Prof. D. D. Eley, M.Sc., Ph.D., 8.30 p.m., Chemical Laboratory, The University, Cambridge. Joint meeting with the University Chemical Society.

Institute of Fuel

November 25. 'Peat-fired Power Stations,' by W. Cronin and J. F. Lang, 5.30 p.m., Institution of Civil Engineers, Great George Street, London, S.W.1.

Institution of Chemical Engineers

November 16. 'Gas Refrigerating Machines,' by J. L. W. Köhler, 4.30 p.m., Science Museum, London, S.W.7. Joint meeting with the Low-temperature Group of the Physical Society.

November 22. 'The Adiabatic Absorption of Hydrogen Chloride,' by T. A. Kantyka and H. R. Hinchlieff, 3 p.m., College of Technology, Manchester.

December 11. 'Synthetic Acetic Acid,' by R. Page, 3 p.m., The University, Edmund Street, Birmingham.

December 11. 'Vapour and Gas Evolution from Low-vapour-pressure Liquids,' by G. Burrows and F. H. Preece, 3 p.m., College of Technology, Manchester.

Graduates' and Students' Section

November 12. 'Feed Back in Chemical Engineering Processes,' by F. F. Ross, 6.30 p.m., Caxton Hall, Westminster, London, S.W.1.

November 13. Joint symposium on the 'Mixing of Solids,' 2.30 p.m., Darlington Technical College.

Incorporated Plant Engineers

November 16. 'Instrumentation and Thermostatic Controls,' Teddington Controls Ltd., 7.15 p.m., South Wales Institute of Engineers, Park Place, Cardiff.

November 25. 'Compressed Air in Industry,' Holman Bros. Ltd., 7.30 p.m., Grand Hotel, Sheffield.

Institute of Petroleum

December 8. 'Design and Operation of a Fluidised Pilot Plant for the Fischer-Tropsch Synthesis,' by C. C. Hall and A. H. Taylor, M.B.E., 5.30 p.m., 26 Portland Place, London, W.1.

Institution of Mechanical Engineers

November 12. 'Inspection, Explosion and Breakdown of Boilers and Pressure Vessels,' by J. Eyers, 5.30 p.m., 1 Birdcage Walk, London, S.W.1.

November 16. 'Measurement of Flow,' 6.45 p.m., 1 Birdcage Walk, London, S.W.1.

Institute of Metals

November 18. 'Aluminium and its Alloys as Engineering Materials,' by E. Elliott, 7.15 p.m., Cleveland Scientific and Technical Institution, Middlesbrough.

November 24. 'High-temperature Water Cooling of Open-hearth Furnaces by Means of Steam-Producing Elements,' by F. J. Feltoe and P. M. Moreton; 'The Cooling of Large Rolling Mill Drives,' by P. M. Moreton; 7.30 p.m., University Buildings, St. George's Square, Sheffield.

CHEMICAL & PROCESS ENGINEERING

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